## **The Fiber Society**

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#### **Conference Chairs**

Heidi Schreuder-Gibson and Phil Gibson U.S. Army Soldier Systems Center Natick, Massachusetts

### **Book of Abstracts - Posters**

U.S. Army Soldier Systems Center (Natick)







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#### **Woven Fabric Engineering Using Artificial Intelligence**

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Engineering of fabric deals with application of science and revealing the relationships between the raw material, process, and the finished product to achieve desired functional or aesthetic effects in the fabric. The success of fabric engineering depends on -

- reliable objective measurements,
- prediction and control of fabric quality and performance attributes.

The application of science over the years has revealed, that the important wearing performance and qualities of apparel fabric namely - comfort, fabric handle, drape, crease and wrinkle recovery, tailorability, and garment appearance - are related to low-stress mechanical properties of fabric such as tensile, extension, compression, bending, shear and fabric surface properties. For applying engineering principles to fabric manufacturing process, precise and accurate quantification of these properties becomes necessary. These fabric properties can be objectively measured, specified and quantified by commercially available instruments such as the Kawabata Evaluation System for Fabrics (KESF) and the Fabric Assurance by Simple Testing (FAST) instruments.

Attempts are being made to design fabrics by using expert systems and neural networks i.e. basically by the use of Artificial Intelligence. The AI system must be capable of doing three things: store knowledge, apply the knowledge stored to solve problems, and acquire new knowledge through experience. Expert systems are engineered to simulate a human expert. But experts in the design of fabrics to meet mechanical specifications are only available in industrial fabric area; where breaking strength, tear resistance etc. are the essential requirements, such expert systems will have little value for manufacturing apparel fabrics to be used for clothing. Even if the experts are available, development, validation and verification of such systems are slow and tedious. Maintenance and up-gradation of the system are also found to be difficult. Artificial neural networks offer a completely different approach to problem solving. They try to provide a tool that both programs itself and learns on its own. Neural networks are structured to provide the capability to solve problems without the benefits of an expert and without the need of programming. They can seek patterns in data that no one knows are there. The main feature that makes neural networks the ideal technology for modeling fabric interactions is that they are nonlinear-regression algorithms that can model high dimensional systems and have a very simple, uniform user interface. These models offer alternatives to traditional concepts of knowledge representation, its acquisition, and its application in processing.

Most of the researchers have used neural networks based on back propagation learning algorithm. In this research, *radial basis function* neural network has been applied to engineer the fabric properties, because of several advantages including design simplicity and easy processibility. Artificial neural networks have the advantage that, once the accurate forward model of the fabric properties is obtained, it can, in turn, be used to develop an inverse model of the relationships, i. e., to predict constructional parameters from the properties of the fabric. In this research, about 100 polyester-viscose blended suiting fabrics of wide variety of constructions were sourced from leading manufactures in India. The fabrics were analyzed for fibre composition, thread linear density, twist in single and doubled yarn, thread density/cm, yarn crimp%, fabric areal density in g/m². From these, fabric warp, weft and total cover were calculated. The fabric properties were measured on FAST set of instruments. The properties

measured were- warp and weft extension%, warp and weft bending rigidities, fabric formability, shear rigidity. Fabrics were also tested for breaking strength and breaking elongations in warp and weft directions.

The inputs to the radial basis function neural network were the important constructional parameters of the fabrics. In all 19 input fabric structural parameters were fed to determine 11 fabric properties, which were the targets to the network. The input-output pairs of the fabric data was randomly divided into 80 pairs of fabric data set for training the network and 16 fabric input-output data set for testing the generalization ability of the network.

The overall prediction error was 13.02%. It was observed that, only 1.14% of the predictions were not as per expectations. There was very good correlation between the actual and the predicted values (r = 0.985). The network could successfully model the structural changes in the fabric to predict the trends in the fabric properties and can be confidently used in designing and development of fabrics as per the desired specifications.

In the second part of the research, the radial basis function neural network was trained and generalized well to predict yarn and constructional parameters of the fabric from its mechanical properties (i. e., inverse model). The overall prediction error was 10.4%. The coefficient of correlation between the actual and predicted values was 0.99. The network successfully simulated the fabric structure in response to changes in fabric properties. All the trends predicted by the network were as per the accepted structure-property relationships of polyester viscose blended fabrics.

These analyses indicate that neural networks could be implemented in expert system to find solution to the fabric designer's queries on fabric structure-property relationships. An expert system was developed using radial basis function based neural network as its knowledge base to construct the design rules. Graphic user interface was provided for easier interaction with the system. Radial basis function has successfully predicted the structure-property relationships of polyester-viscose suiting fabrics. It can be used to optimize a particular fabric property for different end-use applications, knowing the relationships between different parameters that determine the specific fabric property. They can also be used to identify the different levels of combinations of process parameters and material variables that yield the desired property of fabric. From this a specific combination of process and material variables resulting in maximum savings in cost and time can be selected. It is expected that this study would be more useful to characterize technical textiles due to capability of radial basis function neural network for forward and reverse engineering applications.

### Effect of Planar Conduction of Moisture on Measured Water Vapour Permeability of Thin Woven Fabrics

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#### Abstract

In the study, water vapour permeability of six woven fabrics of similar structure but differing in composition and cut into samples of different size was experimentally determined. The objective of this research work was the investigation of the effect of sample dimensions on the results measured by means of the commercial PERMETEST instrument. It was found, that in most cases, the increase of relative vapour permeability due to conduction of condensed moisture in the plane of the measured samples towards the sample edges did not exceed 6%, which confirms the possibility of non-destructive assessment of vapour permeability of garments in specialized shops to verify the declared parameters or in case it is required by a customer.

#### Introduction

Water vapour permeability of fabrics presents, along with fabric thermal resistance, the most important characteristic of clothing comfort. That is why increased attention is paid to this parameter in recent decades [1].

Nevertheless, the published research works are mainly focused on testing of this parameter in official laboratories and the used instruments are generally costly, time consuming and require special samples cut from pieces of fabrics. If these samples are large, their price can increase substantially the total price of each measurement. Moreover, the necessity of specially sized samples avoids the non/destructive measurements on tailored garments, due to high price of the completed products.

The objective of this paper is an analysis of the possibility of the measurement of vapour permeability on large pieces of fabrics or on garments, and analysis of precision of these measurements, if compared with the measurement on cut samples. In the study, water vapour permeability of six woven fabrics of similar structure but made of various materials and cut into samples of different size was experimentally determined. All experiments were carried out with the Czech PERMETEST instrument manufactured by the SENSORA Comp.

#### Description of the PERMETEST instrument used for testing the water vapour permeability

This instrument presents in its design a small "skin model" [2]. Slightly curved porous measuring surface is wetted (either continuously or on demand) and exposed to parallel air flow of velocity 1,5 or 3 m/s. The tested sample is located in the distance of 1-1,5 mm from the wetted area of diameter 80 mm and characterised by high thermal conductivity. The heat flow generated by evapo-ration of liquid water from the measuring surface is measured by a special heat flow sensor integrated into the porous layer. Thus, very low time constant of the whole system was achieved, resulting in short time of measurement – full signal is registered within 2 minutes when measuring the permeability of synthetic fabrics and blends, whereas wool fabric, due to their complicated moisture management, require up to 8 minutes. The whole system can be heated to temperatures exceeding the room temperatures, or can be kept at the room temperature, in order to maintain the isothermal working conditions.

The instrument is not delivered with any climatic device, because its use under standard laboratory conditions (at 22°C and relative humidity 55%) offers reasonable precision of measurement. Results of measurement are expressed in units defined in the ISO Standard 11092.

#### Experimental results and their evaluation

All the samples were woven in plain weave and composed of most 100 % natural and synthetic polymers in grey state. Their square mass varied from 125 to 160 g/m<sup>2</sup>.

The PERMETEST instrument requires samples, which are larger then the diameter of wetted area (60 mm in our case, but at new instruments it was increased to 80 mm), due to fixing the sample in circumferential clamps. To assure the defined dimensions of the permeated area (to simulate the cut samples), the fabric non-permeability was achieved by means of printing of impermeable polymer in the form of circle of diameter 60 mm. Diameters of other circles were 80 mm, 100 mm and the full large sample.

Experimental results in terms of relative water vapour permeability (where 100% presents the "permeability" of free measuring surface) are displayed in the next Fig. 1. All the results present the average values from 10 measurements on each sample. Variation coefficients in most cases did not exceed 5%, which confirms good measurement precision for this kind of measurement [3].

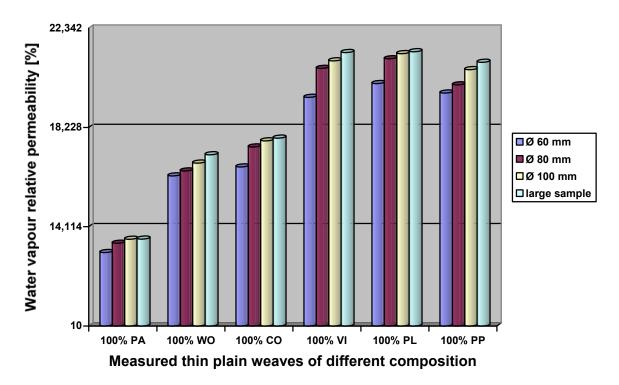


Fig. 1. The effect of sample dimensions on the measured relative water vapour permeability of thin woven fabrics similar in structure but differing in chemical composition

From Fig. 1 follows, that the lowest levels of permeability were found for samples of diameter 60 mm, which coincides with the wetted measuring area of the instrument. If the sample dimensions exceed the measuring area of the instrument, then the determined water vapour permeability increases by 4-9%. This effect can be explained by planar conduction of condensed vapour from the boundary of the measuring area towards fabric edges. Lowest permeability increase (up to 6%), as expected, was found for fabric made of common synthetic polymers and blends.

The determined increase of water vapour permeability is in most cases small enough to permit the non-destructive measurements of commercial textile products in cases when customers require to confirm the properties of their goods or for comparative tests of various garments in specialised shops [4] etc. Nevertheless, this study was focused on thin woven fabrics only. A deeper study will follow, to investigate the water vapour permeability thick and heavy fabrics also.

#### Acknowledgement

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#### Manipulation of Nylon 6 Crystal Structures with Molecular Nanotubes

#### Min Wei and Alan E. Tonelli

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Nylon polymers consist of polyethylene segments (CH<sub>2</sub>)n separated by peptide units (NH-CO) which are packed either parallel or antiparallel in their crystal lattices. These peptide units provide hydrogen bonding between polymer chains, giving nylon some of its unique properties. In contrast to other highly crystalline polymers like polyethylene, nylon polymers can have their degree of crystallinity controlled over a wide range. They also have a unique combination of stiffness, toughness, lubricity, and resistance to abrasion, fatigue, and temperature that makes them among the most versatile thermoplastics in use today. By changing the amide density, one can modify such properties as the melting point, modulus, low temperature impact strength, moisture absorption, and chemical resistance to metal salts and acids.<sup>1</sup>

Nylon 6 is a member of the nylon family and is a versatile polymer. Nylon 6 exhibits polymorphic structures that contain two types of stable crystal forms: monoclinic  $\alpha$ -form and pseudohexagonal (or monoclinic)  $\gamma$ -form (see Figure 1), which are very sensitive to the method of preparing samples. The  $\gamma$ -form nylon 6 crystal is constituted by non-planar polyamide molecules adopting the parallel-chain-arrangement of hydrogen bonding, whereas the polyamide molecules in the monoclinic  $\alpha$ -form crystal are planar and packed in the more stable antiparallel-chain-arrangement of hydrogen bonds. The  $\alpha$ -form is more stable than the  $\gamma$ -form presumeably because of shorter, stronger hydrogen bonds.

Nanotubes, which are fine capillaries with an inside diameter of nm order, have attracted great interest because of their peculiar structures. Recently, a new series of nanotubes with diameters smaller than carbon nanotubes have been obtained by complexing polymers with cyclodextrin molecules in the channel form.<sup>2</sup> In these nanotubular inclusion complexes, the guest polymer chains occupy narrow cylindrical channels (Diameter ~ 0.5-1.0nm) created by the stacking of cyclodextrins in the crystalline lattice. As a consequence, the included guest polymer chains are constrained to assume highly extended conformations and are generally segregated from neighboring included polymer chains by the channel walls of the host crystalline lattice. When these complex crystals are treated to remove the host, but without damaging or dissolving the guest polymers, they are forced to coalesce into a bulk polymer solid. DSC and small-angle X-ray scattering (SAXS) observations of crystallizable polymer samples coalesced from the cyclodextrin nanotubes have often revealed melting temperatures that are elevated from those measured on samples crystallized from their completely disordered solutions and melts, and an absence of discrete reflections from the long spacing between chain-folded lamellae. These observations suggest an extended-chain crystalline morphology for the IC-coalesced polymer samples.

In this study, we successfully formed an inclusion complex between nylon 6 and  $\alpha$ -cyclodextrin and attempted to use the cyclodextrin nanotube to manipulate the polymorphic crystal structures of nylon 6. To prepare the polymer cyclodextrin inclusion complex, nylon 6 was dissolved in a mixture of formic and acetic acids and added to a  $\alpha$ -cyclodextrin dimethyl sulfoxide solution. The mixed solutions were stirred on a hot plate for 2 hours at  $50^{\circ}$ C, and then cooled to room temperature while stirring for another 6 hours. The precipitate was filtered and dried under a heating lamp and in a vacuum oven at  $40^{\circ}$ C to obtain dry polymer cyclodextrin inclusion complex. To remove the cyclodextrin nanotube, the inclusion complexes were placed into HCl aqueous solution (pH=1.0) with heating at  $80^{\circ}$ C. The mixture was stirred for 10 min. and then filtered to remove coalesced polymer. The filtered polymer was washed several times with cool water and then dried in a vacuum oven at room temperature. The formation of nanotubular inclusion complex was verified by Wide Angle X-ray Diffraction (WAXD) and CP/MAS  $^{13}$ C NMR. To investigate the differences between as-received nylon 6 and nanotube treated nylon 6, FTIR, WAXD, CP/MAS  $^{13}$ C NMR and DSC were employed.

Because nylon 6 crystallizes in the  $\alpha$ -crystal form, the  $\gamma$ -crystal form, or a mixture of both, depending on the crystallization conditions used, a reliable determination of the  $\alpha$  and  $\gamma$  contents is important. The FTIR band assignments for both crystal forms of nylon 6 have been reported in the literatures.<sup>3</sup> It is evident that the bands in the region between 1300 and 800 cm<sup>-1</sup> show clear differences between the two crystalline phases. The bands at 928, 959 and 1200 cm<sup>-1</sup> were attributed to the  $\alpha$ -crystalline phase, whereas the band at 973 cm<sup>-1</sup> was attributed to the  $\gamma$ -crystalline phase. In Figure 2, for the as-received nylon 6 chip, there is a strong band at 973 cm<sup>-1</sup>, showing that these samples contain a considerable amount of the  $\gamma$ -crystal form, with a small amount of the  $\alpha$ -crystal form indicated by the weak bands at 928, 959, and 1200 cm<sup>-1</sup>. In contrast, the FTIR spectrum of nylon 6 extracted from the cyclodextrin nanotubes does not show the band at 973 cm<sup>-1</sup>, but strong bands at 930, 959 and 1200 cm<sup>-1</sup>, indicating a much higher fraction of  $\alpha$ -crystal form. Another exciting result is suggested by the absorption peak at 1030 cm<sup>-1</sup>. According to FTIR studies of nylon 6 yarns, it is clear that the intensity of this peak increases with increasing draw ratio. In the FTIR spectrum of the inclusion complex coalesced sample, there is a very strong 1030 cm<sup>-1</sup> peak compared to as-received nylon. This may demonstrate that the

extended, planar conformation adopted by nylon 6 in the cyclodextrin nanotubes is substantially retained after coalescence.

Wide-angle X-ray diffraction is often used to determine the total crystallinity and the ratio of  $\alpha$  and  $\gamma$ -crystalline phases for nylon 6. In the X-ray diffraction patterns of nylon 6, the diffraction peak at  $2\theta = 21.8^{\circ}$  is contributed by the  $\gamma$ -form crystal of nylon and a pair of peaks,  $2\theta = 20^{\circ},24^{\circ}$  are distinctive features of the  $\alpha$ -form crystal of nylon 6. Also there is another broad peak at  $2\theta = 22^{\circ}$  for mesomorphic  $\beta$ -form. Figure 3 shows the WAXD patterns for as-received nylon 6 and cyclodextrin nanotube treated nylon 6. In the pattern of as received nylon 6, it is apparent that there is a fair amount of  $\gamma$  form crystal, although the peak at  $21.8^{\circ}$  is overlapped. However, the  $21.8^{\circ}$  diffraction peak almost disappears in the X-ray pattern of cyclodextrin nanotube treated nylon. For the treated nylon 6, the two strong diffraction peaks characteristic for the  $\alpha$ -form crystal, with much less of the unstable  $\gamma$ -form crystal and amorphous material, can be observed.

Solid-State NMR is one of the more powerful and versatile tools to study polymer structure, morphology and dynamics. The CH<sub>2</sub>  $^{13}$ C resonances of nylon 6 occur in the range of 15-50 ppm vs TMS and overlap strongly. This frequency range includes resonances of the amorphous,  $\gamma$ - and  $\alpha$ -crystalline phases. Resonances of the amorphous phase, which are much broader than the crystalline resonances, are not separately visible. However, the narrower line widths of the crystalline fraction cause them to dominate the CP/MAS spectrum, and the lines assigned here are therefore essentially due to the crystalline fraction. Some methylene carbons have a chemical shift which is sensitive to the crystalline modification, and therefore, a distinction between the different crystalline phases is possible. The peak at 43.9 ppm is assigned to C1 in the  $\alpha$  polymorph and the peak at 41.3 ppm to C1 in the  $\gamma$  form. The CP/MAS  $^{13}$ C NMR spectra of as-received nylon 6 and nylon 6 coalesced from cyclodextrin nanotubes are given in Figure 4. From this figure, it can be seen that the  $\alpha$  form crystal dominates the crystalline region of nylon 6 coalesced from cyclodextrin nanotubes.

DSC was carried out to obtain the thermal and crystallization behaviors of nylon 6 as-received and treated with the molecular nanotubes, although it is not possible to distinguish between the two crystal forms of nylon 6 by this method. Table 1 lists the melting temperature  $(T_m)$ , the crystallization temperature observed upon cooling  $(T_{cc})$ , and the crystallinity of both nylon 6 samples. Compared to the as-received sample, elevated  $T_m$  and  $T_{cc}$  and an almost doubled crystallinity were found for the sample extracted from cyclodextrin molecular nanotubes.

In summary, threading into and extraction from cyclodextrin nanotubes is an effective way to both transform nylon 6 from the  $\gamma$  to its  $\alpha$  polymorph and to obtain a much higher level of crystallinity. The capability of cyclodextrin nanotubes to manipulate the crystal structures of nylons is likely due to the confining channel environment for the included polyamide chains, which requires them to assume highly extended, narrow conformations in order to fit into the stacked cyclodextrin host crystalline channels.

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Table 1. Thermal properties obtained from DSC for as-received and nanotube treated nylon 6

	As received Nylon 6	Nanotube treated Nylon 6
$T_{\rm m}$ (°C)	215.6	219.3
$\Delta H_f(J/g)$	55.8	100.4
Crystallinity (%)	35.1	63.2
$T_{cc}$ (°C)	179.9	180.0
$\Delta H_{cc}$ (J/g)	60.2	66.2
Temperature range of crystallization(°C)	190.7-156.8	188. 6-161.8

<sup>\*</sup> $\Delta H_f$  of 100% crystalline nylon 6 taken as 158.8 (J/g)<sup>5</sup>

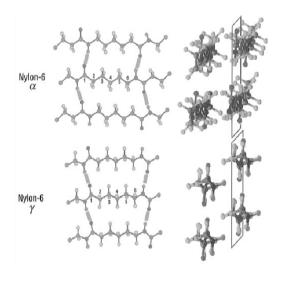


Figure 1. Structures of  $\alpha$  and  $\gamma$  forms of nylon 6

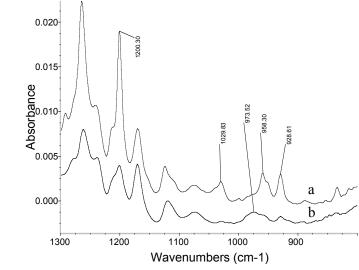


Figure 2. ATR-FTIR spectra of (a) nylon 6 extracted from cyclodextrin nanotubes and (b) as-received nylon 6

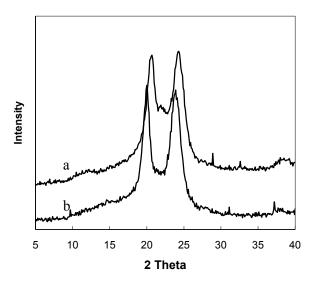


Figure 3. WAXD patterns of (a) as-received nylon 6 and (b) nylon 6 extracted from cyclodextrin nanotubes

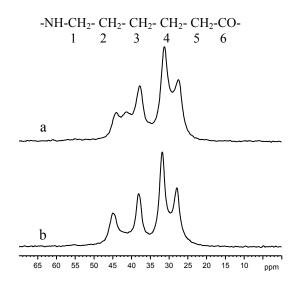


Figure 4. CP/MAS  $^{13}$ C NMR of (a) as-received nylon 6 and (b) nylon 6 extracted from cyclodextrin nanotubes

#### BIO:

Min Wei was born in the PR of China. He has BS and MS degrees in Polymer Chemistry from China, and for the past 3 years has been working on his PhD in Fiber & Polymer Science at NCState under my supervision. He has already co-authored more than a dozen papers, and expects to finish his PhD dissertation in 2003.

#### The Description and Properties of Medical Textiles

#### Nurhan Onar

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Medical textiles means that the textile materials are suitable for usage of all surgery field with together other non-textile materials. Medical textiles can be classified as external and internal medical applications or respectively non-implantable and implantable textiles. Common raw materials for medical textiles are polyester, polyamide, polytetrafluoroethylene, polypropylene, polyurethane and carbon, glass, cotton, viscose, silk, collagen, alginate, chitin, chitosan, polycaprolactan (PCL), elastomer fibers.

In the most case, requirements of medical textiles are; not to be toxic, allergenic or carcinogenic, to withstand sterilization by all known methods, i.e. by steam, radiation or gas etc., a barrier to moisture and bacteria, to have high mechanical properties such as tear strength, burst strength, abrasion resistance, tenacity; to be biocompatible, bioabsorbable; to have the properties of anti-static, water- and moisture- repellent or impermeable, air- and water vapour permeability (breathability), to pass flammability tests, not to allow skin irritation, to be flexible, comfortable, soft, pliable; not to inhibit to movement, not to give rise to sweat or wetness from patient's skin, to be a non-slip surface, to be lint-free, to have optimal costs.

Aims of this research are generally to clarify the textiles which are used in all surgical applications and to summary requirements of medical textiles whilst based on compiling literature.

**Key Words**: Surgical textiles, implantable, breatheability, water-repellent, absorbable, biocompatible, cross-infection, sutures.

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I graduated from the Textile Engineering Department of Ege University that is the oldest and the most successful department for textiles in Turkey. Now, I am continuing MSc degree and I am in the last semester on my MSc degree. I have given presentations on "Medical Textiles", "X-ray, C-NMR and H-NMR spectroscopic analysis methods", "The Usage of Enzymes on Wool Dyeing and Antifelting Processing", "Fiber-reinforcement composite materials", and "The Properties and Test Methods of Geotextile Materials" in the past. Also I am interested in automotive textiles for example airbag systems.

#### Characterization of Electrospun Fibers of Polyimide/ MMT Nanocomposites Zhaohui Sun, Darrell H. Reneker

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Electrospinning is a process using an electrically driven jet to produce sub-micron polymer fibers from polymer solutions or polymer melts. This process has been applied to different kinds of polymers. The diameter of the resulting fibers range from nanometers to micrometers.<sup>1</sup>

In this study, ion exchanged montmorillonite clay was used to prepare polyimide/clay nanocomposites. Polyimide fibers with or without clay were electrospun from the polyimide solutions or dispersion of exfoliated clay in polyimide solutions. The resulting fibers were controllably thinned for observation by TEM and SEM using plasma etching.<sup>2</sup>

This method is not only an effective way to reduce the thickness of fibers by removing polymer, but also a direct way to study the orientation and dispersion of clay in polymer matrix. Montmorillonite layers were observed to be aligned along the fiber axis in electrospun fibers (Fig 1). Electron diffraction patterns of clay were obtained at the same time.

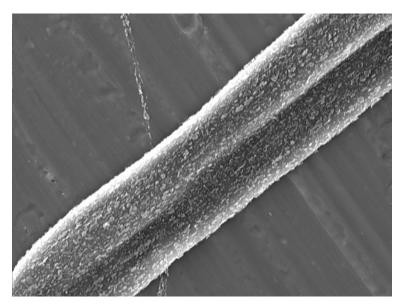


Fig 1. Polyimide /Clay fiber after plasma etching

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## Protective Textiles: Permethrin Treated Camouflage Uniforms Bartley McNally

U.S. Army Soldier Systems Center, Natick, Massachusetts

The Army has long maintained a strong interest in applying the naturally occurring insect repellent, Permethrin, to camouflage uniforms and bednets as a protection for the Soldier against arthropodborne diseases (invertebrate animals with jointed legs and segmented bodies such as mosquitoes, ticks, etc.). The potential for transmission of naturally occurring arthropod-borne diseases such as malaria, hemorrhagic fever, and sandfly fever to the Soldier is enormous if appropriate preventative countermeasures are not immediately instituted upon arrival in endemic areas. In addition, arthropod disease vectors are potential carriers of biological warfare agents and the discomfort factor of high populations of biting insects has a strong influence on Soldier morale and cannot be regarded lightly.

Synthetic pryrethroids (of which Permethrin is a family member) are a relatively new class of insect repellents that exhibit a high toxicity to a broad spectrum of insects while maintaining a low toxicity to mammals. Pyrethroids are lipophilic compounds that are almost insoluble in water. They differ from other lipophilic compounds such as chlorinated hydrocarbons by their susceptibility to attack by metabolic systems. They are therefore biodegradable and will not accumulate in the food chain.

A researcher at Natick developed several methods of application including a factory method of mass treatment of camouflage uniforms and bednets that is at once safe, convenient, cost-effective, and environmentally safe. During 1999 an extensive field-test of mass treated camouflage uniforms was conducted with the 1<sup>st</sup> Battalion, 509<sup>th</sup> Infantry (Airborne) at Fort Polk, LA. Soldiers wearing the treated uniforms were rotated into the field for 2-3 week periods, exposed to all weather, day and night. Extended time was spent lying and sleeping on the ground and the treated uniforms were worn an average total of 55 days for an average of 20 hours per day.

Presented below are the key findings of the field-test\*\*.

Does the Permethrin-treated uniform offer equivalent or superior protection over what is currently used by the Soldiers in the field?

Yes Ninety percent of the soldiers felt that they had fewer bites than usual when wearing the treated uniform.

## Does the Permethrin treatment have any impact, either positive or negative, on the health of safety of the evaluation participants?

**No** Ninety-four percent (94%) of the soldiers at the end of the evaluation felt that the treated uniform was safe to wear. While there were some reports of irritation that respondents attribute to the Permethrin treatment, this was only true for about one-fourth of the survey group. Most of these problems were minor (a sensation or low-level rash) and were transitory (about a week in duration). Laundering the uniform a few times seemed to eliminate the problem for half of those experienced it, while others reported that it faded over time.

## What impact does the laundering and exposure to field conditions (i.e. sun, rain, dirt, etc.) have on the effectiveness of the Permethrin treatment?

**Minimal** The treated uniform probably lost some of their effectiveness over time. However, 90% of the Soldiers felt that they were still receiving fewer bites when they were the treated uniform at the end of the evaluation.

## What impact, either positive or negative, does the Permethrin treatment have on overall soldier performance and effectiveness in the field?

**Positive Impact** It appears that the mass treatment method of Permethrin application probably did improve soldier performance to some extent. These Soldiers felt that wearing the treated uniforms led to fewer insect bites, controlled insects on and around them, and offered better protection than an untreated uniform. Furthermore, the factory treated uniform received significantly higher satisfaction ratings for suitability for field use and overall performance than the untreated uniform.

During the past 2 years West Point cadets have experienced a high incident of Lyme disease from indignant deer ticks. Consequently, in April of this year West Point officials ordered all camouflage uniforms worn by the cadets and those to be issued to the cadets in the future be treated with Permethrin by the factory method developed at Natick.

\*\*User Evaluation of Factory Treated Permethrin Field Uniform, Customer Feedback Team, SBCCOM, Natick, 8 May 2002.

#### The Role of Baffles in Fiber Dispersion in Mixing Tanks

#### H. Vahedi Tafreshi\* and Behnam Pourdeyhimi

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Dispersion and separation of fiber bundles requires exposing them to a shear stress field to overcome inter-fiber frictional forces. To this end, fiber-mixing tanks are equipped with baffles to enhance the shear and agitation in the water to help disperse the fiber bundles. The time and the agitation required to separate and disperse the fibers is dependent on the fibers being used. It is well known however, that excessive agitation will give rise to the formation of rope defects in the output because of the high-energy vortices that form behind these baffles. Optimizing the baffle geometry and position is therefore, of critical importance in the wet-lay process.

The current paper discusses a series of numerical simulations to model fiber dispersion inside the wet-lay mixing tanks. Turbulent flow field is obtained by solving the Navier-Stokes equations in a two-dimensional geometry. The turbulent features of the flow are captured using RNG *k-e* model. The effects of the baffles and their orientation with respect to flow streamlines are simulated.

Desirable mixing condition is the one that has a uniform distribution of shear stress and/or turbulent kinetic energy to agitate the fibers without having vortex and dead-water regions. In this regard, we studied five different mixing tanks each of them having different baffle shape/position. First case (case A) is a four-flat-plat baffle tank with the agitator placed at the tank center. Second case (B) is similar to the case A but has a different baffle position. In case C we used the same baffles as in the case A but employed an off-center agitator. To study effect of baffle surface area in enhancing the shear field inside the system and eliminating large vortices at the same time, we designed streamlined baffles presented in case D. The last configuration is case E which takes advantage of the concept of increasing baffle surface area introduced in case D in addition to its spiral baffles.

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Once the fiber-free water field is obtained, fibers are introduced to the solution domain from three point-sources placed in different locations inside the tank. Particles are tracked inside the tank for a sufficiently long time. In this study fibers are modeled by spherical rigid particles having the same volume as the fibers. Particles have a density of 1.38 g/cm<sup>3</sup> and a diameter of 110 micron representative of 6 mm PET fibers with 1.5 denier linear density.

Our results indicate formation of recirculation dead-water zones behind and in front of flat-plat baffles (cases A, B, and C). We showed that the dispersed fibers may get entrapped in these regions and cause rope formation due to highly swirling flow. The baffle configurations in case D and E are such that they do not permit the formation of large circulation zones and, therefore, probability of rope formation should be decreased. However, turbulence intensity and kinetic energy have been seen to be lowest in cases D and E. This indicates that logs in cases D and E have more chance to stay unbroken by the time they leave the tank as compared to other cases. Comparing case D and case E, we showed that the spiral form of the baffles in case E directs the flow toward the periphery of the tank and increases the average flow velocity and turbulence near the walls while having a fairly good strain rate. We therefore, concluded that the latter case considered (E) has a superior operation over the others.

#### Presenting author's biography:

Hooman received his BS and MS in Mechanical Engineering in Tehran, Iran. He then moved to Lappeenranta, Finland where he obtained his PhD in Energy Technology. He served as Research Associate at the University of Milan, Italy for two years after his graduation. Hooman has joined Nonwovens Cooperative Research Center, North Carolina State University since October 2001 and is involved with simulation and experiment on the hydroentangling waterjets.

#### Physician's Attitudes Toward the Utilization of Smart Fabrics in Cybermedicine *Ella Carter*

University of North Carolina-Greensboro

In recent years, smart fabric innovations have received a great deal of attention in textile industry publications as well as consumer market publications. One smart fabric category that has received much publicity is the one that focuses on smart fabric medical innovations. Although smart fabric is a generic term that includes a variety of "high tech" or high performance fabrics including computational, digital, interactive, electronic and sensory textile, the smart fabric product category designed for medical use includes a wide range of products. Some of the fabrics use fiber optic components while others function utilizing special polymers that are woven or knitted together with conventional fabrics to produce a wide range of touch-, pressure- or temperature sensitive products. Others utilize a special thermal process that enables to fabric to become electro- or thermally conductive while still others combine normal and electroconductive textiles with thin composites allowing fibers to react to changes in electrical current, body chemistry and temperature. The resulting fibers are fully compatible with the functionality of the base material to give the look and feel of a normal fabric despite unique properties ranging from the full integration of digital and analogue functions ranging from voice synthesis, remote control, warning signals, telecommunications to patient diagnostics as well as a list of other stateof-the art medical uses.

Although smart fabrics medical innovation represent a major advance in textile innovations, many of the applications are still limited to military environments. However, the technology is slowly diffusing into mainstream medical and consumer markets and as it diffuses smart fabric researchers and marketers are searching for ways to understand the diffusion process and factors influencing the technology acceptance process.

The objective of this on-going work is to investigate physicians' attitudes toward the utilization of smart fabrics in cybermedicine and to create a conceptual framework for explaining how three constructs: perceived risk, ease of use and usefulness affect physicians' intention to use smart fabric technology for diagnostic purposes and in cybermedicine. Physicians' attitudes toward the

utilization of smart fabrics for these purposes will ultimately influence the diffusion process of smart fabric medical innovations since physicians acceptance is key to social acceptance of smart fabrics for use in medicine, whether as an instrument to further their efforts to monitor patients through cybermedicine or through actual utilization of smart fabrics for diagnostic purposes.

This research is designed to assist smart fabric marketers in understanding the technology acceptance process, further the status of diffusion research and assist in identifying key determinants associated with the diffusion of smart fabric medical innovations.

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Ella Carter is a third-year doctoral student at the University of North Carolina-Greensboro, majoring in Textile Products Design and Marketing. She has extensive experience in marketing, advertising, has worked as a consultant to a number of major corporations in the US and has conducted international textile and apparel research. In addition, she has taught Contemporary Issues in Textile and Apparel at the University of North Carolina-Greensboro and has taught marketing seminars for start-up businesses. She is a VF Corporation Fellow and has received the Nancy J. Sears Award for Academic Excellence. She holds and MBA in New Venture Management from the University of St. Thomas in Minneapolis/St. Paul, MN and a Bachelors of Art in Art and English-Journalism/Communications from Averett College in Danville, VA.

## Mechanical Properties of Electrostatically Spun, Nonwoven Fiber Membranes A. Pedicini and R.J. Farris

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An electrostatic field was applied to solutions of commercial polyurethane thermoplastic elastomer. Electrospun fibers of various polyurethanes—Pellethane $^{\text{TM}}$  and Estane $^{\text{TM}}$ —were collected as non-woven isotropic membranes 0.1-0.2mm thick, for mechanical characterization. Solutions were prepared in solvent of low vapor pressure, creating a relatively wet spinning fiber jet and resulting in a "fused" fibrous membrane that can be tested with conventional mechanical methods.

Mechanical properties of the electrospun fiber membranes are compared to the bulk properties of the respective TPU. Bulk specimens were prepared by compression molding thin plaques from pellets received from the manufacturer. Standard ASTM 1708—which does not allow for computation of elastic modulus—was followed to determine tensile strength and elongation to break of electrospun and compression molded samples. Elastic moduli will be determined by dynamic mechanical testing.

The thermoplastic polyurethanes electrospun include Pellethane 2103-70A, Pellethane 2103-80AE (Dow Chemical Co.) and Estane 58277 and Estane 58237 (Noveon, Inc.). In each case, dimethyl formamide was the solvent used. The electrospinning set-up consists of a glass pipette, 1mm stainless steel wire (positive lead), and aluminum foil target (grounded). Electrospinning voltages ranged from 10-15kV. Polymer solution concentrations ranged from 7%-10%wt. polymer in DMF. Scanning electron micrographs were recorded using a JEOL 35CF Scanning Electron Microscope. Tensile tests were performed using an Instron 4468.

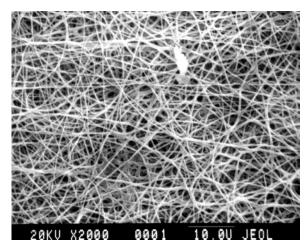


Figure 1. Electrostatically spun Pellethane 2301-80AE fibers.

Initial results from tensile tests of electrospun TPU fiber membranes show a marked decrease in elongation to fracture. The most characteristic difference between electrospun and conventional specimens is the shape of the stress-strain curve produced by each. Compression molded samples show stress-strain behavior typical of elastomeric polymers. The electrospun specimens, however, have near linear shaped curves.

In general, the electrospun membranes have densities approximately 1/3 that of the bulk samples. Assuming an effective load-bearing cross-section for the electrospun membranes of 1/3 that of the bulk specimens, the electrospun membranes have tensile strength and modulus comparable to the bulk samples. The reasons for these qualitative and quantitative differences will be investigated and presented.

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#### **Education and Experience**

#### 1999-Present University of Massachusetts-Amherst Doctoral Candidate

• Studying electrospinning of polymer solutions, producing sub-micron diameter fibers and mechanical characterization of electrospun fiber mats

1999-2000 University of Massachusetts-Amherst Master's Degree

**1998 University of Kentucky** Inducted as a member of Sigma Alpha Mu, International Materials Engineering Honor Society

1996-1998 University of Kentucky Research Assistant

• Worked in the laboratory of Dr. Lynn S. Penn, performing single-fiber composite tests to evaluate fiber-matrix interface quality of polymer-matrix composites

## 1997 Summer Research Experience for Undergraduates (REU Program), Center for Membrane Sciences, University of Kentucky

- Developed a high strain-rate test for single-fiber composite interface characterization 1994-1998 University of Kentucky Undergraduate studies
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#### **Publications**

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#### **Fatigue Behavior of Nylon Industrial Yarns**

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#### **ABSTRACT**

The purpose of this research is to develop a fracture mechanics technique suitable for industrial nylon fibers. Specifically, a J-Integral technique will be developed and used to predict the strength limitations of these fibers. Successful research has already been done on larger fibers using the critical energy release rate for linear elastic materials ( $G_{Ic}$ ). However, the fibers in the current study are much smaller and a significant amount of plasticity precedes failure. Thus the nonlinear critical energy release ( $J_{Ic}$ ) rate will be determined in this research, which will help predict the critical flaw size that dictates the strength of the nylon industrial fibers. There are five phases in the development of this technique:

- 1) Devise a technique suitable for tensile and cyclic loading of small fibers
- 2) Develop the J-R curve which describes the material resistance to crack advancement
- 3) Fatigue individual fibers in an effort to increase the existing flaw (crack) size
- 4) Perform tensile tests on the fatigued samples of various crack lengths to obtain J
- 5) Plot J,  $J_R$  vs. crack length to determine the critical flaw size and  $J_{Ic}$

At the conclusion of this research, manufacturers in the domain of fiber development will have better insight as to how individual fibers should be processed in order to obtain certain mechanical properties. For example, the tire industry can use this research to develop more durable and lightweight aircraft tires. This is directly proportional to cost savings for the airline industry, since a lower amount of fuel will be needed for operation.

#### METHOD OF RESEARCH

A technique has already been established for testing these small fibers. This involves attaching the fibers to a 1" tab and using a cylindrical sub-mount to prevent slippage. The next step is to prepare a J-R curve, which is a material property that describes the resistance to crack propagation. To prepare this resistance curve, ASTM recommends using a multiple specimen technique. To achieve this, several specimens of the same initial crack length will be loaded to various crosshead displacements and the area under the curve will be measured. The specimen will then be loaded to failure to expose the fracture surface and SEM (scanning electron microscope) will be used to determine the initial flaw size. Each specimen will constitute a different point on the  $J_R$  vs. crack length curve and as a general rule the data will be fit to a power law of the form [1]:

$$J_R = C_1 \Delta a_p^{C_2}$$

The next step in this research will be to fatigue individual fibers for different cycle times in order to propagate an existing flaw in the specimen. This method is known as "precracking," and different crack lengths will be obtained through precise control of the cyclic loads. The expression that relates the crack growth per cycle can be obtained through a power law expression as described by Dowling and Begley [2]:

$$\frac{da}{dN} = C\Delta J^m$$

An Enduratec mechanical tester (ELF 3200) will be used for the cyclic testing. It has the capability of cycling up to several hundred hertz and can also be used for tensile testing.

After "precracking," tensile tests will be performed on the specimens in an effort to determine driving force curves (J) versus crack length (a). The equations that govern the nonlinear energy release rate J is described as:

$$J_{TOT} = J_{el} + J_{pl}$$

$$J_{TOT} = \frac{K_I^2 (1 - v^2)}{E} + \frac{\eta_p U_p}{B(W - a_0)}$$

This expression gives the nonlinear energy release rate in terms of the plastic energy absorbed by the specimen,  $U_p$ . This energy is directly proportional to the area under the load-displacement curve and can be solved by the following equation [3]:

$$U_P = \int_0^{\Delta_P} P d\Delta_P$$

Since all other terms are known, the total nonlinear energy release rate (J) can be plotted as a function of applied load for various initial crack lengths a. A relationship can then be made between  $J_{TOT}$  vs. a. The critical flaw size can be predicted by plotting  $J_R$  and  $J_{TOT}$  vs. a. The point at which the curves are tangent describes a critical point in the system and further loading leads to unstable crack growth.

This gives a critical nonlinear energy release rate for the fibers as well as a critical flaw size that leads to unstable crack growth. This is very useful for manufacturers, since certain spinning processes can be modified to eliminate flaws of this critical size and the strength of the fiber will be increased dramatically.

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Dry-jet Wet Spinning of Fire Safe Polymers Poly(hydroxy amide) and Poly(methoxy amide)

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In the history of fibrous non-flammable materials, poly-p-phenylenebenzobisthiazole(**PBZT**) and poly-benzoxazole(**PBO**) have been of major interest since Toyobo and Dow started to produce **PBO** fiber with tenacity of 5.8 Gpa in 1994. Poly benzimidazole(**PBI**) was the most prominent fibrous fire-safe material for various purposes in the textile area before the appearance of **PBO** and **PBZT**.

Although **PBO** fiber has excellent strength, the strongly acidic solution-dope for fiber spinning is a major disadvantage[1-3]. The poly(hydroxy-amide) family(**PHA**s), the precursor of PBO, is a good non-flammable material itself and turns into PBO under high temperature producing small molecules such as water or alcohols. The produced small molecules depress heat transfer and improve the non-flammability of the polymer. **PHA**s are dissolvable in aprotic solvents such as N, N-dimethyl acetamide (DMAc), N-methyl pyrrolidone(NMP) and N, N-dimethyl formamide (DMF) rather than strong acids.

Among **PHA**s, poly(hydroxy-amide)(**PHA**) and poly(methoxy amide) (**PMeOA**) show fairly low heat release capacity. Our research group has studied these two homo-polymers and the copolymer in terms of the non-flammability and processibility. **PMeOA** shows a galss transition temperature occurring before demethylation or decomposition. In previous work, the melt spinning of **PMeOA** was attempted and melt spun fiber was attained, although it has poor physical properties.

In this work, **PHA** and **PMeOA** fibers were spun by dry-jet wet spinning from PHA/DMAc and PMeOA/DMSO spinning dope solutions. The spinnability of PHA/DMAc solution is good at **PHA** 5wt% concentration and **PMeOA** fiber was spun at 30 wt% solution. To investigate the effect of coagulant on the solidification process of fiber, the coagulant power,  $\xi$ , and the mass transfer rate difference,  $\Delta K$ , were evaluated in various coagulants: water, methanol, ethanol, and i-propanol. The gelled model filament technique was used for obtaining the value of  $\Delta K$ . Consequently, the order of  $\Delta K$  and  $\xi$  of the coagulants depends on the molecular size of each coagulant.

**PHA** and **PMeOA** fiber was spun in an ethanol coagulant by dry-jet wet spinning. The spun **PHA** and **PMeOA** fibers show a tensile strength of 350 MPa and 82 Mpa, a maximum strain of 10% and 9.5%, and a Young's Modulus of 17.6 Gpa and 2.6 Gpa, respectively.

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## Biodegradable/Bioresorbable Scaffolding for the Re-Growth of Bone Tissue Developed from Electrospun Fibers

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The goal of this project is to develop scaffolding from polycaprolactone (PCL), a biocompatible polymer that is also biodegradable/bioresorbable. Blended with the PCL will be hydroxyapatite (HA), a mineral found in natural bone. HA is an osteoconductive material that has shown excellent biocompatibility and the ability to form chemical bonds with natural bone. The scaffolding will be characterized by SEM and TEM. Following osteoblast cell growth, SEM will be used determine the extent of penetration and cell viability within the scaffolding. If successful in this project, the scaffolding would serve as a device to re-grow bone within the body.

The scaffolding specimens were formed by electrospinning. A PCL/HA solution in acetone was formulated and electrospun onto an aluminum plate, resulting in a matrix of PCL fibers surrounded by beads of HA and PCL. The ultimate processing goal is to electrospin a substantially thick 3-D structure that has enough void space to allow the cells to infiltrate the entire structure

The research goals are; development of the scaffolding through the electrospinning process, characterization of the scaffolds with SEM and TEM prior to seeding of the cells; and examination of the extent of penetration and viability of the incubated cells using SEM. TEM will be utilized in characterizing the electrospinning samples, examining fiber size, shape and networking. SEM will gather information on fiber size and shape and the presence of ceramic deposits as well as determining the extent of cell penetration into the scaffolding. Once all of the analysis has been completed it will be determined if electrospinning is a viable process for the formation of scaffolding, and if the scaffolding formed by this technique is capable of supporting cell growth.

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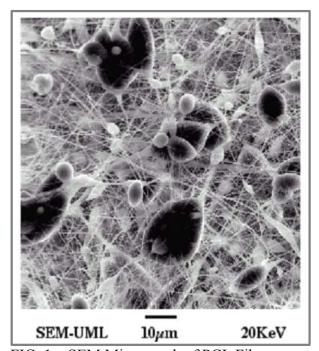


FIG. 1 – SEM Micrograph of PCL Fibers Surrounded by Beads of HA and PCL (1000x)

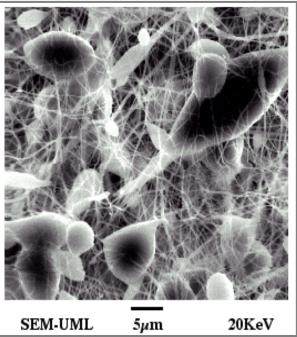


FIG. 2 – SEM Micrograph of PCL Fibers Surrounded by Beads of HA and PCL (2000x)

#### Stephen Iadarola

I am currently a graduate student in the Chemical and Nuclear Engineering Department at the University of Massachusetts Lowell. In 2001 I received a bachelor of science degree in Chemical Engineering from same university. My research interests include biomaterials, medical device design, and polymers. I am planning to complete my Master's thesis in the fall of 2002.

#### **Tridimensional Fabrics for Smart Textiles**

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Textile materials are everywhere in people's day life, either at the present, as well as in the past. One cannot think about life of people without associate it to textiles. Probably, because of this, we may not perceive how textiles influence our quality of life. This influence has increased from past to present and everything at a world scale.

Textiles are much more than the primary satisfaction of the need to cover the body, even than the satisfaction of presentation need, this is, the aesthetical pleasure and influence. Textiles must be more and more a source of health, comfort, security, healthy environment, economic added value, culture

Thus, the improvement of Man and Textiles relationship goes through the development of new multifunctional materials, the search of new and/or better application solutions and the optimisation of manufacturing processes.

One of the most modern research subjects relates to smart textiles, searching the performance optimisation of garments during wear, adding to them other functionalities, which are important to the quality of people's life, but not feasible before. In terms of product engineering, this is presently possible, either with special fibres and structures, and by incorporating non textile materials in fabrics and/or clothes

The main objective of smart textiles is to add comfort and security to the people. Concerning comfort, it can be referred to temperature and moisture conservation adequate to the body. In respect to security, it comprises, for example, the acquiring of vital signals in critical situations, such as the third age people, security professions and military actions.

A difficulty associated to smart textiles relates to ergonomic comfort. As a matter of fact, fabrics and knits designed to hold special textile and non textile materials for the improvement of thermal comfort and signal acquisition, loose ergonomic comfort, this means, they become quite uncomfortable when wearing them.

Taking into account the usefulness of functionalities that smart textiles can add, it is fundamental to develop textile structures to hold special materials, exhibiting a similar mechanical behaviour as compared to the conventional ones. In this way, the objective of this work is to contribute to the development of multi-layer warp and weft fabrics, presenting similar bending and shear rigidity and formability values to those of the most commonly used military fabrics.

The application of multi-layer fabrics in smart textiles is due to the need of hiding the special yarns, which add functionalities to clothes, to avoid being uncomfortable and non aesthetical. Thus, the smart textiles must have an appearance similar to that of a simple fabric used in clothing, although developing behind the regular functionalities, other specific ones. In order to

keep the weight/m<sup>2</sup> and the mechanical behaviour both similar to those of simple fabrics, the multi-layer ones must be built with thinner and less twisted yarns and with other patterns than plain weave. Among others, an example of weave used in the tested fabrics which gives them a normal 2/2 twill fabric appearance, both in face and reverse sides is the following:

	f	r	f	S	r	f	r	f	S	r
r	٠	+	٠	٠		٠		٠	٠	+
S	٠					٠		٠	0	
f										
r	•	+	•	•	+	•		•		
f			-	0		•				
r	•		•	•	+	•	+	•	•	
S	•		•	0		•				
f			-							
r	•		•			•	+	•	•	+
f									0	

f – face yarn; r – reverse yarn; s – special yarn.

A good way to characterise the ergonomic comfort of fabrics is through its mechanical performance, mainly in what concerns bending and shear rigidities and formability. These fabric mechanical properties are evaluated using Kawabata and/or FAST (Fabric Assurance by Simple Testing) systems. In the later one, these properties are defined by the following equations:

```
Bending rigidity (B), (\muN·m):
B=9,81·10<sup>-6</sup>·W·c<sup>3</sup>
W – weight/m<sup>2</sup> (g/m<sup>2</sup>); c – bending length (mm).
Shear rigidity (G), (N/m):
G=123/e(5)
e(5) – extensibility at 5gf/cm (%).
Formability (F), (mm<sup>2</sup>):
F=B·[e(20)-e(5)]
14,7
e(20) – extensibility at 20gf/cm (%).
```

Tests were made in conventional and multi-layer fabrics using FAST system, the results achieved being quite satisfactory, this is, the ergonomics of multi-layer and simple fabrics being close to each other.

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### Molecular Simulation of Polymer Crystallization: Nucleation from Pre-oriented Melt

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Molecular dynamics simulations are performed using a united atom force field for polyethylene in order to characterize the homogeneous nucleation and its enhancement in the presence of strong orienting flow stresses. The simulations are carried out in NPT ensemble, with periodic boundary conditions in x, y and z directions. Nucleation of crystallites in the quiescent melt is too slow to observe for flexible chains like polyethylene, unless such rates are artificially enhanced through the use of stiff torsional potentials. However, nucleation can also be accelerated by orienting the material on the molecular scale, in accord with experimental observations. Using this approach, we have prepared pre-oriented melts of 20 C<sub>400</sub>H<sub>802</sub> chains by applying stresses to the melt in the x-direction ( $\sigma_{xx}=1\times10^8$  Pa,  $\sigma_{yy}=1\times10^5$  Pa and  $\sigma_{zz}=1\times10^5$  Pa) at 425 K and allowing the system to evolve for 10's of ns. Using this oriented melt as a starting configuration, we cool the system to several different temperatures below the crystallization temperature and observe nucleus formation. Our simulations have been performed using instantaneous quenches with  $\sigma_{xx}=1\times10^5$  Pa,  $\sigma_{yy}=1\times10^5$  Pa and  $\sigma_{zz}=1\times10^5$  Pa at 250 K, 300 K and 350 K, respectively. The number and size of nuclei are investigated by convoluting the orientation order tensor with position. The different degrees of relaxation experienced by the simulation cells are observed, and the greatest relaxation occurs at the highest temperature. A different number of nuclei are formed in each cell, and the highest number of nuclei occurs at the lowest temperature. Also apparent is that the resulting crystals are both larger and less aligned with the orientation direction at the highest temperature.

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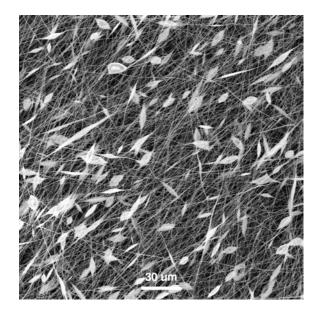
B. S., Fiber and Polymer Science, Seoul National University, 1995

## Crucial parameters for electrospun polymer nanofibers Zeng Jun, Michael Bognitzki, Haoqing Hou, J. H. Wendorff, Andreas Greiner

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Electrospinning is a straight-forward method to produce nano-scaled polymer fibers, which attracts increasing scientific and commercial attention due to the versatile applications of nanofibers in many fields, such as catalysts, sensors, biomedical applications, filtration, templating. Recently, electrospun polymer nanofibers were successfully applied as templates for the preparation of a variety of tubes by the so-called TUFT process<sup>1-3</sup>. The application of electrospun nanofibers as templates requires precise knowledge how shape and dimensions of fibers are controlled by process parameters. Therefore, we have empirically investigated electrospinning of a wide variety of polymer systems, which will be the topic of our presentation.

The fiber shape can be influenced by many parameters, e. g. solvent vapor pressure, surface tension, solution viscosity, solution conductivity, and glass transition of the polymer. In our experiments, a systematic investigation on each of these parameters and their respective effects on fiber shape was performed. It was found that solution viscosity (SV) is a key factor responsible for fiber morphology. With higher SV produces, smooth and thick fibers were formed, whereas with lower SV, thin but beaded fibers were formed; solution conductivity (SCond) had effect on the size and morphology of fibers. Increase of SCond resulted in fine and smooth (no beads) fibers. Numerous kinds of additives were used in our experiments have persuasively proved this effect. A representative example for the effect of additives on the shape of electrospun polylactide (PLA) fibers is shown in Fig. 1. Surface tension (SFT) was considered as a driving force to form beads or beaded fibers. According to this, reduced SFT should favor formation of smooth fibers and increased SFT for formation of beaded fibers. However, our results show for PLA that SFT is of minor importance for fiber shape.



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Fig. 1: 2% PLA in Dichloromethane without additive

2% PLA in Dichlormethane with 0,45% additive

# Acknowledgement:

The authors are indebted to BMBF for financial support.

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# Water vapor transfer through textile under a temperature and humidity gradient Nefzi Nada, Ben Nasrallah Sassi

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Moisture movement in a porous medium is a process that occurs in many engineering applications. These include moisture movement in soils, nutrient uptake by plants, drying of food, and petroleum extraction. This process is also of a special interest to the textile industry. The working mechanisms of water vapor transmissions through textile fabrics are not well known. The rate at which they are able to transmit water vapor is measured under realistic condition. An apparatus has been constructed to measure the water vapor resistance of these fabrics and to examine the effect of a temperature and humidity gradient on moisture vapor transport.

Experiments involve relative humidity of 40% 50%, and 70% and temperature of 30°C. Clothing is an integral part of human life, and an understanding of the role of clothing in the thermal balance of the human body and thermal comfort under steady-state conditions has developed over the past. Even in very cold weather, active people can over-heat. The body often has to sweat in order to balance heat loss with heat production at a comfortable body temperature.

To have a feeling of comfort, the individual must have the skin dries. The moisture emitted by the human body must be evacuated. Humans rely on the evaporation of sweat to remain comfortable and prevent overheating in hot environments and during exercises. In some situations, the evaporative rate from wet skin is less than the rate of sweat secretion. The rate at which clothing can transmit sweat is not well known.

In order to quantify the comfort of a clothing, it will be interesting to understand the phenomenon of heat and mass transfer through textile.

Water vapor permeability of materials can be measured with a guarded hot plate by saturating the plate surface with water. The power required to maintain the surface at a given temperature is related to the rate at which water evaporates from the surface of the plate and diffuses through the material. Woodcock developed a moisture vapor permeability index known as  $i_m$  which serves as a convenient relative measure of the moisture vapor permeability of materials. The  $I_m$  value is a relative measure of permeability of the material to the passage of water vapor. J.T.Finn and al adopted this method to study the effect of imposing a temperature gradient on moisture vapor transfer through water resistant breathable fabrics. The value of the

clothing vapor resistance is usually derived either from measurements of samples of the clothing materials, Farnworth and Dolhan , measured on complete clothing ensembles on a wetted thermal manikin, McCullough and al, or determined on human subjects, Havenith and Lotens . Van Beest and Wittgen described a modification with greater facility and accuracy, which permits the variation of RH at the sample from saturation to about 35%.

The objective of this research is to measure the rate at which textile fabrics transmit water vapor and to examine the effect of a temperature and humidity gradient on moisture vapor transport. An experimental apparatus has been developed to simulate the evaporation of the sweat through textile fabric and study its diffusivity. Water is heated to 34°C in order to simulate as sweating body, and the rate at which the vapor is transmitted through these fabrics is measured.

A humidity and a temperature gradient are imposed to create a driving force able to cause the moisture vapor migration of the wettest area towards the least wet area. This thus amounts creating a diffusive flow through the sample.

For the purpose of our investigation, we consider Polyester/ Cotton, Polyester, Viscose fabrics. We study effect of ambient humidity and temperature on vapor diffusivity.

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# Fabric Softness Classification Using Surface Fiber Profiles: Fuzzy Logic Approach

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Fabric softness is one of the most important attributes determining the quality of garment. Objective criteria for fabric softness, however, have not been successfully established yet mainly due to the system's insensitivity to surface fiber profiles. In this study, we use statistical and fuzzy-based heuristic models and the surface fiber statistics such as coefficient of variation (CV%), mean deviation (MD) in order to classify softness of wool fabrics. The results have been compared to both the KES data and human subjective evaluations. 19 wool fabrics examined for surface measurement have been evaluated by trained personnel for tactile sensation. The surface characteristics have been captured and analyzed by using image processing procedures, and proper correlation analysis have been performed in order to determine the important surface statistics affecting subjective evaluation for softness. Two separate models have been employed in order to classify the fabrics into 3 groups, "Low", "Medium", and "High" degree of softness. A statistical model, Fisher's discriminant function, has been developed by using 10 wool fabrics. The parameters were statistically estimated and evaluated in terms of significance and variability. A series of cross validation experiments has been made for the rest of the fabrics. The results show that, the fuzzy logic based- model outperformed the statistical model in both classification accuracy and consistency.

#### **Image Analysis System**

The original images obtained have been processed by a thresholding algorithm into the images of surface contours. Being converted into binary formats, the images have been further processed in order to obtain the statistics such as M, CV, MD, RN. Figure 1 compares an original image of surface fibers and its corresponding contour image in a photographic format.

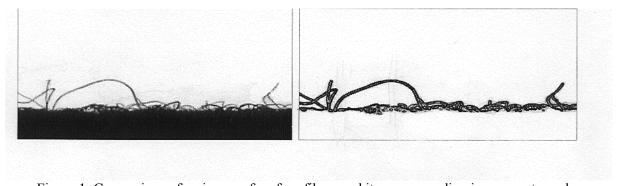


Figure 1. Comparison of an image of surface fibers and its corresponding image contoured

### Relationship between Surface Fiber Profiles and Subjective Evaluation

It has been found from some preliminary experiments that there is a strong correlation between surface fiber profiles and corresponding subjective evaluation scores. Figure 2 shows that a clear statistical evidence of the relationship mentioned above.

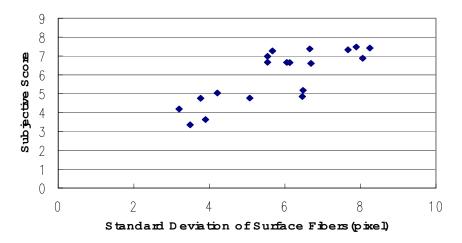


Figure 2. Relationship between surface fibers and subjective evaluation

#### **Discrimination Function for Fabric Softness Classification**

Stepwise discriminant analysis has been performed in order to examine the relationship between the surface characteristics and the tactile sensation. Four descriptive statistics have been used for discrimination, which are mean (M), coefficient of variation (CV), Range (RN), mean deviation (MD). The resulting discriminant function is represented by

-4.159 +0.089\*M+0.194\*CV-0.030\*RN-0.437\*MD with a misclassification of 11.1%.

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# **Electrospinning Biocompatible Polymer Nanofibers**

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Electrospinning produces fibers with diameters in the range of nanometers to microns in a short time. More than a hundred polymers were electrospun in our lab. These fibers have a high surface area per unit mass, which is greater than that of ordinary textile fibers. Nanofiber are used in filters, protective fabrics, advanced composites, enzyme support in bioreactors, agriculture, wound dressing, biosensor, scaffold and other applications.

The electrically charged surface of a droplet of polymer solution initiates a fluid jet. The jet path is initially straight, in the direction of the electric field. An electrically driven instability develops expanding spiral loops. The self-repulsion of the charge carried by the jet makes each element of the jet grows larger. As the solvent evaporates, the fluid solidifies to fibers and deposits on the collector. The behavior of an electrospinning jet can be observed by using high-speed camera.

Many polymers such as polycaprolactone, polyurethane, polylactide-co-polyglycolide acid, polyethylenimine are electrospun to biocompatible polymer nanofibers. Polymers that dissolve in the same solvent are electrospun easily, forming nanofibers with separated phases as shown in figure 1(a). Sol-gel solution, and mixture of polymer with crosslinking agent also electropsun to crosslinked fibers as shown in figure 1(b)-(d). Small insoluble particles and additive liquid that were dispersed to the solution were electrospun in nanofibers. Those particles and additives were encapsulated in the dry nanofiber. Polymer nanofibers and nonwoven mats of nanofibers provided the matrix that supports such additives. Optical and electron microscopes were employed to characterize the electrospun nanofibers.

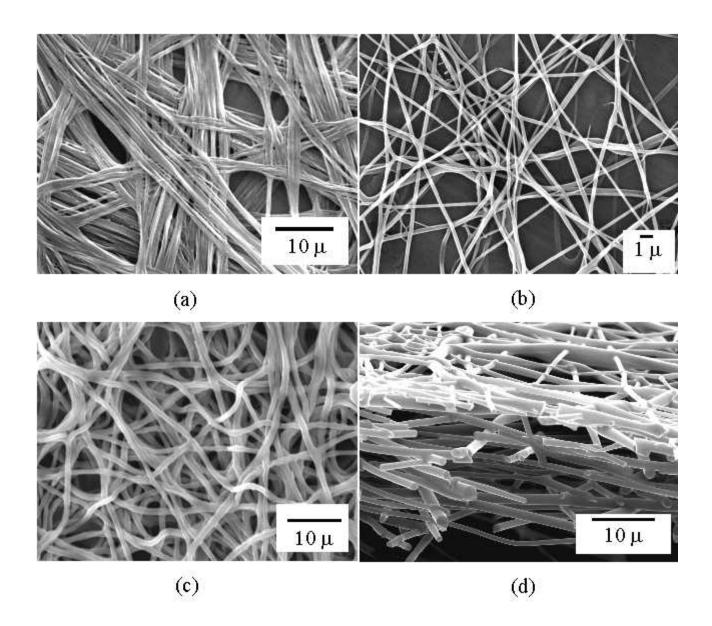


Figure 1. Images of scanning electron microscopy of electrospun (a) polycaprolactone, (b) polyvinylamine, (c) polyethylenemine, and (d) silicon oxide (from sol-gel solution) nanofibers

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# Poly(meta-phenylene isophthalamide) nanofiber templates for nanotube synthesis *Wenxia Liu, Edward A. Evans,*<sup>†</sup> *Darrell H. Reneker and Brian J. Satola*<sup>†</sup>

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#### Abstract

Sacrificial polymer fibers can be used to create freestanding polycrystalline nanotubes of many materials. Electrospinning provides a straightforward method to produce fibers with diameters in the nanometer range. The scientific basis for the mechanism of fiber formation, the physical properties of nanofibers, as well as applications of nanofibers in the areas of filtration, composites, biomedicine, and electronics has been investigated. There is interest in developing processes for larger scale production. Polymer nanofibers were used as sacrificial templates for nanotubes of metals and semiconductors. <sup>1-3</sup>

In this work, poly(meta-phenylene isophthalamide) (MPD-I) fibers (Nomex<sup>®</sup>) were chosen for their high thermal and mechanical stability. MPD-I fibers are stable up to 300 °C or higher, which allows the use of a wide variety of vapor deposited materials without melting or degrading the polymer. MPD-I nanofibers were prepared by an electrospinning process. Nanotubes were prepared by first coating MPD-I nanofibers with aluminum and then removing, by thermal degradation and solvent dissolution, the template polymer fiber. This process is a candidate for making tubes of any material that can be deposited as a continuous film. Such tubes can be used directly or coated with other materials.

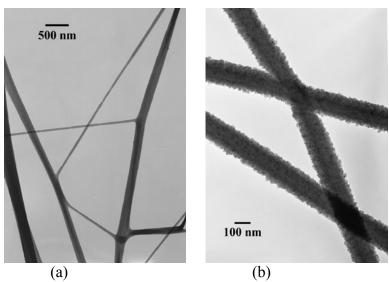


Figure 1 (a). TEM image of as-spun MPD-I nanofibers. (b). TEM images of aluminum-coated MPD-I fibers.

Figure 1 (a) is a transmission electron microscopic (TEM) image of the MPD-I nanofibers. The fiber surface was smooth and featureless. The fibers were coated with aluminum films by an RF sputtering process. Figure 1 (b) shows aluminum-coated MPD-I fibers.

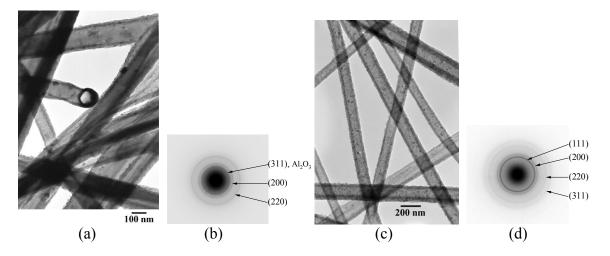


Figure 2 (a). TEM image of Al<sub>2</sub>O<sub>3</sub>/Al nanotubes prepared by pyrolysis. (b) Electron diffraction pattern of nanotubes prepared by pyrolysis. (c) TEM images of Al tubes prepared by solvent dissolution of MPD-I fibers. (d) Electron diffraction pattern of nanotubes prepared by solvent dissolution of MPD-I fibers.

Nanotubes of mixed aluminum oxide and aluminum were produced by pyrolytic degradation of the MPD-I nanofiber cores [Figure 2 (a)]. The aluminum coating layers underwent a limited degree of oxidation during pyrolysis of the MPD-I. The aluminum coating layers did not change when the template fiber was removed by dissolution. The average inner diameter of the tubes was around 100 nm. The smallest observed inner diameter of a tube was around 25 nm. The wall thickness of the tubes was in the range from 10 to 100 nm.

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# **Electrospinning of Molten Polycaprolactone in Air**

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The electrospinning process is a simple method to produce nanofibers from polymer fluids and has been under extensive studied <sup>1,2</sup>. Electrospun nanofiber has a promising potential in several applications including filtration, wound dressing and biomaterials application.<sup>3, 4</sup> Few studies, however, had been done with polymer melts <sup>5,6</sup>.

In this study, pellets of Polycaprolactone (PCL) in a glass spinneret were melted with a conductive heat source. Charges were supplied by a metal wire immersed in the molten PCL. A collector plate made of an aluminum sheet was placed 20-40 cm away from the spinneret and maintained at an attractive electrical potential. The electric field strength between the cup and the collector sheet was varied from 25-200 kV/m. Under the applied electric field, a pendant droplet of molten polymer started to deform from a semi-spherical into a conical shape. A droplet of molten polymer was pulled out, trailed by a charged jet that flowed toward the collecting plate where it was collected. The motion of the molten PCL jet was followed and recorded with high frame rate camera. The behavior of the charged PCL jet during electrospinning process can be described into the 3 categories:

- (i) Straight charged jets that flowed toward the collecting plate without the development of any bending instability. The jet often deformed via a buckling mechanism as it landed. As more fibers were collected, they exerted a repulsive force on the incoming jet, which pushed the jet outward as it landed.
  - (ii) Jets that developed the bending instability close to collecting plate.
- (iii) Jets that developed the bending instability in flight. The amplitude of the instability was usually much smaller than that of solution electrospinning. Each segment of the jets developed the bending instability independently (as shown in Fig.1), as a result, each segments experienced different elongation deformation, which lead to a fiber diameter variation.

Only one bending instability cycle was observed with high frame rate video camera. However, SEM images indicated that the charged jet of molten polymer underwent several cycles of bending instability. The diameters of the fibers produced ranged from 200 nanometers to 30 micrometers. Fiber diameter of samples that developed the bending instability tended to be smaller than those without the instability.

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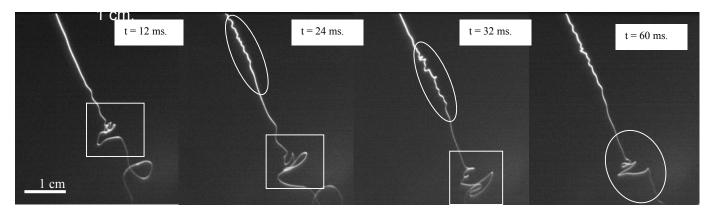


Figure 1: Development of electrically driven bending instability of PCL melt in air

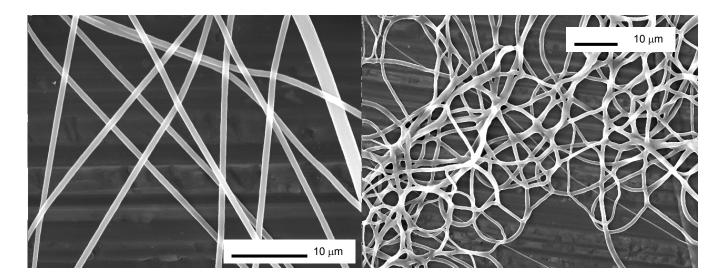


Figure 2: SEM images of PCL fibers electrospun in air

### Prediction of electrospinning jet diameter via interference colors

### Han Xu\*, Daniel Galehouse, Darrell Reneker

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During the electrospinning of polymer solutions, interference colors similar to those seen in soap bubbles can be observed under proper illumination. The colors appear both in the straight part of the jet and in the loops formed by the electrically driven bending instability. The colors depend on the diameters of the spinning jets.

To calculate the scattering, the jet was assumed to be an infinite cylinder. Light impinging perpendicular to the axis is scattered according to Lord Rayleigh's theory of scattering [1]. For a monochromatic incident beam, angular power distributions were calculated for different jet diameters (Fig. 1.). For a chromatic incident beam, spectral distributions were calculated as a function of scattering angles (Fig.2). At a fixed scattering angle, the calculated spectral distribution scattered from the jets were integrated with color-matching functions of  $\overline{x}(\lambda), \overline{y}(\lambda), \overline{z}(\lambda)$  for red peaked at 700nm, green at 546.1nm and blue at 435.8nm, proposed by International Commission on Illumination (CIE) [2]. The integrated result denoted by X,Y,Z gave the weight of red, green and blue in the spectral power distribution of scattered light. X, Y and Z define a color in the CIE color space. The two dimensional CIE chromaticity diagram was created by projecting the 3D space to the X+Y+Z=1 plane. Jets of selected diameters were spotted on the diagram for particular scattering angles. Each spot represents a color, which is the characteristic of the scattering jet as would be observed by normal human eyes.

Laser diffraction was used to precisely measure the diameters at different positions along the jet. A 6% polyethylene oxide water solution was used as a test sample. Jets from  $2\mu$  to  $11\mu$  were measured along a 13.5mm unbent segment, starting 6mm below the spinneret. The jet profile was calculated. Interference colors of the same jet, under xenon arc illumination were observed. The measured diameters and observed colors were compared with the calculated results of the computer model.

This method provided a straightforward way to monitor jet diameters during spinning and related to the fiber size in the bending area. Data extracted from this experiment will provide a firm basis for understanding and controlling the bending, capillary, and other instabilities observed during electrospinning.

<sup>[1]</sup> Lord Rayleigh, On the Electromagnetic Theory of Light, The collected Optics Papers of Lord Rayleigh, Part A. 1869-1892, Optical Society of America, Article 74, 518-528.

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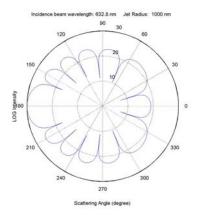


Fig. 1. Calculated angular power distribution of red laser scattered by a  $2\mu$  diameter jet. The laser is incident from the right at 0 degree.

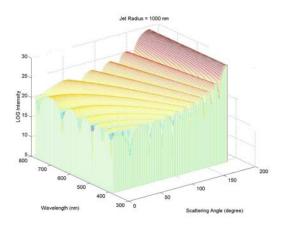


Fig. 2. Calculated spectral power distributions of visible light (wavelength, 380nm to 780nm) scattered by a  $2\mu$  diameter jet.

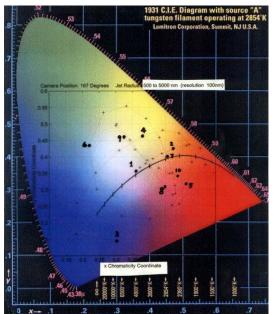


Fig. 3. Interference colors of cylindrical jets with diameters from  $1\mu$  to  $10\mu$  increasing with steps of  $0.2\mu$ . Numbers on the plot are jet diameters in microns.

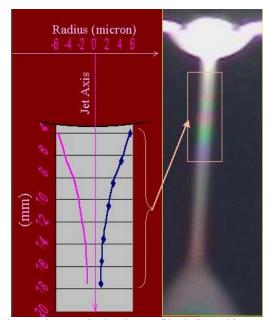


Fig. 4. Electrospinning jet profile (left) and its interference colors (right). Using 6% polyethylene oxide water solution.

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# Surface Modification of Natural Fibres by Using β-Cyclodextrin Derivative Bojana Voncina, Nataša Majcen, Alenka Majcen le Marechal

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Surface modification of cellulose fibres by the fixation of supramolecular compounds which can form inclusion complex with different guest components was performed. For this purpose the natural  $\beta$ -cyclodextrin was used. The most important properties of cyclodextrin may be the capability to form inclusion compounds where the host component admits a guest component into its cavity without covalent bonding. Natural cyclodextrins are widely used in commercial pharmaceutical and food products, recently, also used in laundering and deodorising applications.

In our research an attempt was made to modify  $\beta$ -cyclodextrin by using 2,4,6-trichloro-s-triazine. The reactive  $\beta$ -cyclodextrin derivative was covalently fixed to nucleophilic substrates by a condensation reaction. This new type of surface modification means a permanent transfer of cyclodextrin properties to the treated materials. Analogues to reactive dyes the modified cyclodextrin can be fixed to the textile fibres by well known methods and with common equipment.

Modification of  $\beta$ -cyclodextrin to the reactive specie was followed by HPLC, UV-VIS and FT-IR spectroscopy. The reaction was carried out at the neutral conditions at the temperature of 2 to 4°C for three hours to prepare dichloro-s-triazine derivative of  $\beta$ -cyclodextrin (DCT-cyclodextrin) and further at 50°C at pH 8 for 10 hours to prepare monochloro-s-triazine derivative (MCT-cyclodextrin).

MCT-cyclodextrin reagent was applied to the cellulose fibers without prior purification by a process comprising dipping in aqueous solution of reagent, squeezing, drying, heating and rinsing.

The modified textile materials were characterized by FT-IR spectroscopy, and further some physical properties, such as tensile strength, wrinkle recovery angle were measured. The accessibility of textile materials treated with  $\beta$ -cyclodextrin derivative for guest compounds was tested by vapor method and by spraying the fragrant component onto the material.

In this way modified cellulose fibers could be of interest for home textiles and, when medicals are used as guest compounds, for medical textiles, as well.

### Curriculum Vitae (Bojana Vončina)

#### **EDUCATION**

1991-1995	Colour Chemistry and Dyeing Department, University of Leeds, UK, PhD in Colour			
	Chemistry			
1987-1991	Chemistry and Chemical Engineering, University of Ljubljana, Slovenia, MSc in Organic			
	Chemistry			
1979-1983	Chemical Engineering, University of Maribor, Slovenia			
	BSc in Chemical Engineering			

#### **WORK EXPERIENCE**

1983-1986	analytical chemist at the Quality control department of Lead mine, Mežica, Slovenia,		
1986-1987	researcher at the Textile Institute Maribor, Slovenia		
1987-1991	researcher at the Chemistry Engineering Department, University of Maribor,		
1991-1997	researcher at the Mechanical Engineering Department - Institute of Textile Chemistry,		
	University of Maribor,		
1997	Associate professor at the Mechanical Engineering Department - Institute of Textile		
	Chemistry, University of Maribor		

#### **PROJECTS**

- Eco Friendly Finishing of Cotton Fibres, beneficiaries: IBI, Textile Industry Kranj
- Formaldehyde in textile interlinings, beneficiaries: ZVEZDA, Textile Industry Kranj
- Bilateral cooperation with the University of Leeds, Colour Chemistry Dept.
- Bilateral cooperation with the University of Budapest, Dept. of Organic Chemical Technology, Hungary
- Bilateral cooperation with the University of Nebraska-Lincoln, USA

# Use of Electrospun Nanofibers for Aerosol Filtration In Textile Structures

#### Heidi Schreuder-Gibson and Phil Gibson

U.S. Army Soldier Systems Center, Natick, Massachusetts

The U.S. Army Natick Soldier Center has been studying the enhancement of aerosol protection of fabrics that are coated with a fine layer of electrospun nanofibers. We have electrospun nanofibers onto elastic melt blown liners and investigated the effect of uniaxial and biaxial deformation upon filtration and flow of these nonwoven fabrics. Reported in this paper are the air, water vapor, and aerosol transport properties and pore size measurements of elastic, deformable fabrics. Aerosol filtration decreases with increasing strain for elastic liners, but a clear advantage is observed for electrospun nanofiber-coated fabrics.

Thickness of the electrospun coating has also been found to affect aerosol filtration for nanofibers covering the open pores of fabrics and foams used in protective clothing. Current military fabrics that are used for chemical protection are based upon absorptive layers of carbon-filled foam or layers of carbon spheres bound to a fabric (Saratoga fabric). Elastic as well as non-elastic Nylon nanofibers were applied to the surfaces of these protective layers to measure the effect of nanofiber thickness upon aerosol filtration efficiency in the unstrained state.

Electrospun fibrous membranes are highly porous structures that can be produced from a number of polymer/solvent combinations. Pore sizes ranging from 0.1 to 6  $\mu$ m in diameter can be produced from solvent electrospinning. Air resistance and aerosol protection measurements on experimental electrospun fiber mats compare favorably with properties of textiles and membranes currently used in protective clothing systems.

Two elastomeric electrospun membranes, a melt blown, and a knit Spandex® fabric, were tested under biaxial strain conditions for the transport properties of air flow resistance and aerosol filtration. Air flow resistance of all three materials decreased as the pores were stretched open under increasing strain levels. Liquid expulsion porometry provided a measurement of mean pore sizes under the same conditions of increasing biaxial or uniaxial strain. There was a general correlation of mean pore size with the air flow resistance measurements.

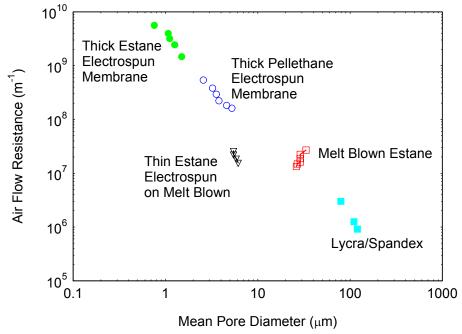


Figure 1. Variation of air flow resistance and mean pore size at various strain levels for five elastic porous materials.

Electrospun nanofiber coatings were applied to open cell foam and the Saragota carbon bead fabric system. Aerosol filtration of 99.9% was achieved with an added nanofiber weight of  $0.5 \text{ g/m}^2$  onto the open cell foam, while air flow resistance through the foam increased by a factor of 10 to approximately the same flow resistance of a typical protective shell fabric.

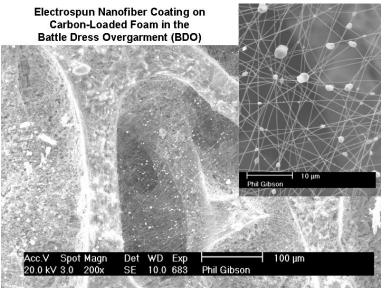


Figure 2. Aerosol particle filtration of electrospun nylon fibers applied to surface of activated carbon-loaded polyurethane foam used in chemical protective clothing.

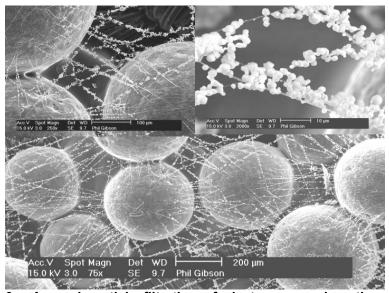


Figure 3. Aerosol particle filtration of electrospun polyurethane fibers applied to surface of activated carbon sphere filter bed layer used in chemical protective clothing.

This study has shown that it is possible to tailor pore size, air permeability and aerosol filtration of elastic nonwoven media by applying very light weight layers of electrospun elastic fibers to the coarser webs. It has been shown that significant deformation of the elastic webs increases air flow, and it might be possible to design controlled flow filters or air bags that are modulated by pressure drop across elastic webs with correspondingly variable porosities.

Biographical Information:

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Ph.D. Polymer Scientist, University of Akron

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Dr. Schreuder-Gibson is a research polymer chemist at the U.S. Army Natick Soldier Center. An employee for the Department of Defense for the past 22 years, she developed solid rocket propellants for the Air Force in the 1980s. Since 1990, she has worked on protective textiles and plastics for the Army Soldier Center, including high strength fibers for armor, biodegradable plastics for food packaging, new flame-resistant materials, and new breathable membranes for protective clothing.

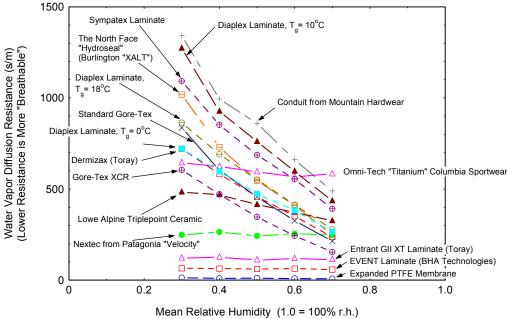
# Temperature-Dependent Water Vapor Diffusion Through Shape-Memory Polymer Laminates: Comparison with Other Waterproof-Breathable Laminates

#### Phil Gibson

U.S. Army Soldier Systems Center, Natick, Massachusetts

The U.S. Army evaluates waterproof-breathable membrane laminates for various environmental protection applications. The standard rainwear material used by the military consists of a three-layer Gore-Tex membrane laminate. The U.S. Army has recently been evaluating the performance of a class of materials known as "Shape Memory Polymers" (SMP). SMPs are polyurethane films that have a glass transition temperature (Tg) tailored to be in a specific range of environmental and body temperatures. When laminated to a fabric, the resulting material is waterproof, windproof, and allows the passage of water vapor. It is claimed that SMPs undergo a transition near the Tg, which results in an increase in water vapor permeability of the polyurethane membrane due to enhanced micro-Brownian motion [1-2]. SMP materials evaluated in this study include Diaplex polyurethane membranes from Mitsubishi Heavy Industries, and Dermizax polyurethanes from Toray Industries. Dermizax also comprises the membrane incorporated into the commercial laminate trade-named "Membrain" from Marmot Mountain, Inc.

Figure 1 shows the baseline water vapor diffusion resistance for a variety of commercial breathable shell fabrics and laminates. Materials that have lower resistance are more "breathable." Testing was done at 30°C. The particular test method used allows one to separate concentration-dependent permeability from temperature-dependent permeability [3].



(Average of Humidity on Both Sides of Sample, Equivalent to Equilibrium Sorption Isotherm)

Figure 1. Water vapor diffusion resistance of SMP membrane laminates and comparable materials.

Figure 2(a) shows water vapor flux as a function of temperature for a subset of the materials shown in Figure 1. Over the temperature range of 5°C to 40°C, the expanded polytetrafluoroethylene membrane (ePTFE) is the most breathable, followed by the EVENT laminate (an ePTFE laminate), and then Gore-Tex XCR. Many of materials are fairly equivalent -- this includes Standard Gore-Tex, the Diaplex laminates, Dermizax, etc. The materials which were less breathable were the Sympatex laminate, and the SMP Diaplex laminate with a  $T_g$  of 10 °C. The results are plotted on a log plot to help show the transitions in permeability that are claimed to occur with SMP membrane laminates. If there were a transition in water vapor permeability at some temperature, it would show up best in a plot such as Figure 2. None of the materials tested showed any indication of being more or less permeable at various temperatures. The water vapor flux simply increases proportionally to the vapor pressure of water as the temperature goes up. The slope shown for all the materials is only due to the variation of water vapor pressure with temperature, and not to any special variation in the permeability of the membranes or laminates.

A possible explanation for misinterpretation of temperature-dependent permeability for SMP laminates is evident in the experimental procedures cited in claims of temperature-dependent permeability [1-2]. Flaws in test methodology, combined with a failure to test standard or control materials alongside the SMPs have apparently resulted in mistaking the vapor-pressure temperature curve present in the experimental results for actual changes in polymer permeability at different temperatures. An example of the type of plot which is often mistaken for temperature-dependent permeability is shown in Figure 2(b), which is simply a few of the materials shown in Figure 2(a) replotted without the log scale on the y-axis.

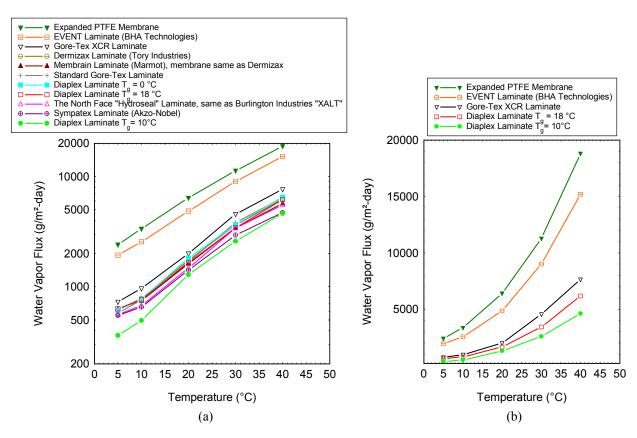


Figure 2. (a) Water vapor flux as a function of temperature for Shape Memory Polymer laminates as compared to various commercial waterproof breathable membranes and laminates. (b) Possible explanation for misinterpretation of experimental temperature-dependent water vapor permeability common in the literature.

Temperature effects are much less important than concentration-dependent effects in the hydrophilic polymer membrane laminates, most of which are based on water vapor permeable polyurethanes. Observed changes in water vapor flux at different temperatures are primarily due to the relationship between temperature and the saturation vapor pressure of water, and not to intrinsic changes in polymer permeability. Shape Memory Polymer films show no special increase in permeability as compared to other waterproof breathable materials. The SMP laminates are comparable to standard Gore-Tex, so they are fairly functional in terms of being "breathable", but they don't have any unique behavior with regard to permeability at different temperatures.

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- 1989 M.S. in Mechanical Engineering, University of Washington
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Conducts studies to measure heat and mass transfer through porous materials. Conducted research on ballistic protection, including thermoplastic composites for helmets. Managed research projects to develop and evaluate novel chemical protective uniform materials. Conducted research in the area of blast protection (transmission of shock waves through materials and their interaction with the human body). Conducted research on detonation and combustion of solid rocket propellant and high-energy explosives while serving as an officer in the U.S. Air Force.

# **Comfort Properties of Textiles as a Function of Treatment**

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#### 1. INTRODUCTION

A soft handle, good appearance and some other surface properties accompanied by appropriate mechanical properties represent the basis for a high quality fabric. The very special characteristics of viscose fabrics, handle and aesthetics are conditioned by the use of high twisted viscose yarns. For production of crepe georgettes fabrics (fabrics made of high twisted yarns) filaments with 1500 - 2300 twists are usually used. Due to the specific particularities of high twisted yarns some modifications of the conventional technological process for the fabrics production must be undertaken. To achieve suitable surface properties, better crease resistance and dimensional stability of fabrics made of cellulose fibres the durable press finishing with different resins is the usual way. The fabrics mechanical and comfort properties are often influenced by the process of finishing therefore the influence of treatment conditions on the fabric handle was studied.

The correct combination of fashion, functionality and feeling (meaning touch) is the most important characteristic for the end user. It is known that the objective evaluation of the fabrics handle is very pretentious. The method for determination of the fabrics handle properties developed by Kawabata is based on the following terms:

- Koshi elasticity and stiffness. It is a parameter of handle where bending strength predominates. High density fabrics and fabrics produced of elastic yarns mainly show this component of handle.
- Numeri smoothness. A feeling of handle involving a few components, including the flexibility, smoothness and softness.
- Fukurama fullness and softness. It reflects a combination of fabrics fullness and elastic characteristics regarding compression and thickness. This parameter is in a close connection with the feeling of the fabric like fabric warmth.
- Sofutasa soft handle. This character includes Koshi, Numeri and Fukurama and it gives a very important common feeling of the fabric. Mainly it represents the smoothness of the fabric.
- Kishimi scrooping feeling. The feeling and sound associated with some lightweight silk fabrics.
- Shinayakasa flexibility/softness. A feeling of softness and flexibility characteristic of many silk fabrics.

#### 2 EXPERIMENTAL

### 2.1 Preparation of the samples

Viscose filament fabrics made of high twisted yarns were studied. Three different fabrics of different origin were compared. All experiments were performed with plain woven fabrics. Viscose - crepe rayon of 133/30/1700 dtex was used. Additionally the finishing process was performed to achieve better end – use properties. Fabrics were resin treated to achieve better dimensional stability and a better crease resistant effect. The fabrics were immersed in the treating solution of Quecodur CW, Gadalan LV 74 and Finistrol TL 806 with different resin concentrations and passed through squeeze rolls to give a wet pick up of 100%. The fabrics were suspended in a force draft oven and pre-dried at 110°C for 5 minutes. After curing for 30 seconds at 170°C the efficiency of the crosslinking was tested by wrinkle recovery angle measurements.

#### 2.2 Analytical methods

Mechanical and physical properties of the grey and the finished fabric were tested according to the standard test norms. Stress – strain curves were obtained on the dynamometer Statigraph S Textechno (DIN 53857, ISO 5081). FAST and KES – F system were used for determination of the fabrics handle.

#### 3. RESULTS

We compared the tenacity and the elongation of fabrics before and after the treatment in the warp and weft direction. The obtained results are within the technological requirements and enduse norms even in the case when viscose samples are crosslinked. Due to the fabrics origin some differences between their mechanical properties are observed but never the less they are not significant.

Untreated fabrics weight between 95 and 118 g/m², they have a bending rigidity of 14 - 19 mm in the warp direction and 12 - 16 mm in the weft direction. Shear rigidity of the sample before finishing is 15-23 N/m, thickness of the fabrics, measured at 0.196 kN/m² 0.26-0.36 mm and 0.21-0.28 mm at 9.81 kN/m². Surface thickness is 0.05-0.07 mm and after relaxation 0.06-0.1 mm. Samples express a lower relaxation shrinkage in the weft direction (0.1-3.7%) compared to the warp direction (2.6%) and 0.6%. Extensibility in wet state is 0.05% and 0.05% in the warp direction and 0.07-0.09 in the weft direction, respectively. Some changes were observed due to the finishing process and fabrics provenience. Additionally physical properties of the samples were determined by means of KES-F system and a general estimation of fabrics handle was given.

As it was expected shari handle component is especially pronounced by viscose crepe fabrics of different producers as this handle characteristic is mainly connected with high twisted fabric constructions. Crosslinking of cellulose increases it. Grades of other handle parameters are in the middle of the scale (approximately 4-5), with the exception of fukurami and kishimi where the values are lower (2-4). Stiffness of the fabrics is not influenced by the treatment conditions while flexibility, fullness and softness of fabrics with a higher content of resin decreases, nevertheless the variations between samples treated with different concentrations of Quecodur CW are not extensively expressed.

#### 4. CONCLUSIONS

In the article three different fabrics are compared. The samples were produced from viscose high twisted yarns. In spite of the very similar fabric construction the fabrics produced by different producers differ slightly regarding the physical properties and thereby also in handle properties. Furthermore a unique model of the influence of the treatment conditions on the surface characteristics changes could not be set.

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Making Matrix-Free Spectra® Fiber Reinforced Composites

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It is proposed that high-pressure high-temperature sintering coupled with thermoforming of Spectra<sup>®</sup> woven cloth can produce multilayer ballistic protective shields. Three important processing parameters are temperature, pressure and time. Relative high temperature near or slightly above the normal melting point is needed to provide sufficient surface melting of the fibers to fuse them together. High pressure is required to constrain the fibers from shrinking or losing orientation and to also consolidate the material. Time is critical for heat transfer and to achieve adequate adhesion between fibers but with minimum loss of original strength. Therefore, this research was conducted to optimize the processing conditions. After examining the properties of the products processed under different conditions by DSC, WAXS, SEM, impact tests, T-peel tests and flexural tests, an optimal processing window was determined. Preliminary ballistic test results have shown that samples made from this method performed slight better than those that are made by conventional methods using the same fabrics with a matrix. It has been demonstrated that it is also possible to shape and mold the fabrics using proper heating and stretching sequences. This essentially matrix-free approach to make high performance composite materials can be utilized to make pressure vessels, high strength tubes, and artificial hip joints, etc. Other polymeric materials could also be processed in similar fashion to make unique products.

# Effect of Fabric Construction on Mechanical Behavior of Fabric Reinforced Rubber

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Fabrics coated with rubber have wide applications in fields such as medical substrates, protective clothing, and flexible membranes for civil structures, airbags, geotextiles and industrial fabrics. As the market for coated fabrics expands to applications with more complex geometries and loading conditions, a competitive edge can be gained by optimizing the selection of fabric substrate and coating materials.

Shear tests, tensile tests and different microscopy methods are used to understand the interaction between the fiber structure and the rubber coating. These results improve our ability to predict and prevent undesirable behaviors such as wrinkling, distortion and tear.

This work includes experimental studies of the effect of various parameters such as fabric construction (e.g., yarn size, weave pattern); shear rate, and coating thickness on coated fabric mechanical response. A controlled set of woven polyester fabrics, with constant warp yarn size and varying weft (or fill) yarn size, as well as different weave patterns (plain weave, 4-harness satin and 8-harness satin weave) are tested.

Table 1. Fabric Parameters

Warp Yarn Type	2 ply /150 denier polyester, 40 epi		
	KOSA Type 787 polyester		
Fill Yarn Types	220 denier		
	500 denier		
	1000 denier		
	Plain Weave (PW)		
Weave Patterns	4 Harness Satin Weave (4HS)		
	8 Harness Satin Weave (8HS)		

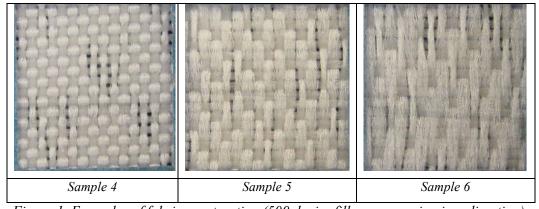


Figure 1. Examples of fabric construction (500 denier fill yarns running in x-direction): Sample 4, Plain weave; Sample 5, 4 harness satin weave; Sample 6, 8 harness satin weave

Figure 2 shows the experimental setup for measuring the fabric shear behavior. The trellis frame shear fixture is designed to apply a pure shear condition to the fabric. Figures 3 & 4 show preliminary shear results for uncoated and coated fabrics with the same size yarn and different weave pattern.

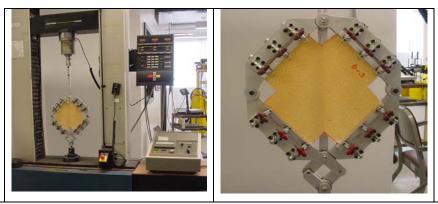
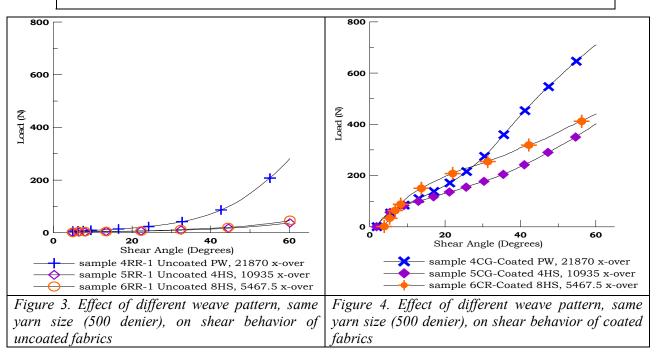


Figure 2. Test set up & the trellis frame fixture for measuring fabric shear response

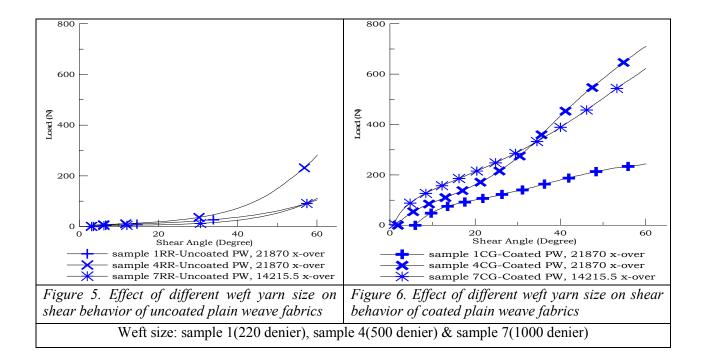


As shown in the Figure 3 because of the tightness of the plain weave, the yarn movement is restricted. This restriction leads to a higher shear load response compared to the satin weaves. However, the difference between weave patterns for the coated fabrics shear behavior is not as apparent as for uncoated fabrics. In the coated fabrics, the movements of yarns in all three weave patterns is dominated by the coating and the behavior is closer to the shear response of pure rubber.

Figures 5 & 6 show the shear behavior of uncoated and coated fabrics with the same plain weave pattern, but with different size weft yarns. Samples 1, 4, and 7 have the same plain weave pattern, but different numbers of crossovers and yarn size, which affects the shear responses as well.

Although the shear response of coated fabric is highly influenced by the rubber, the effect of yarn size and number of crossovers is observed.

Therefore, the effects of weave pattern, number of crossovers and yarn size within the uncoated and coated material is critical. These three distinct areas will be discussed in detail in this paper.



# Biography:

Samira Farboodmanesh is a graduate student at University of Massachusetts Lowell in the Mechanical Engineering Department. She joined the Advanced Composite Materials and Textile Research Laboratory (ACMTRL) in January 2001, as a graduate research assistant. She received her B.S. in Textile Engineering, majoring in Fiber Science and Textile Chemistry, from Tehran Polytechnic University.

Currently, she is working on two different projects. The first project is *Effect of Fabric Construction on Mechanical Behavior of Fabric Reinforced Rubber*, funded by the National Textile Center (NTC), which is also her thesis topic and the second project is *Controlling the Morphology and Orientation of Electrospun Nanofibers*, funded by the National Science Foundation (NSF). Both of the topics involve close collaboration with the Plastics Engineering Department at UMASS Lowell. Ultimately, she hopes to get her Master's degree and pursue her education further more.

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**Treatment of Cotton Fibers with Purified Cellulases** 

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Cellulases find use in a wide variety of ways in the textile industry. Cellulases are used to alter the surface properties of cotton fabrics in processes such as denim stone washing, biopolishing, and are also incorporated in commercial detergent formulations. *Crude* cellulases or commercial cellulases are a mixture of endoglucanases, exoglucanses and beta-glucosidase, with each component having a different mode of action.

Adsorption of cellulase components on cellulose is a key step in enzymatic hydrolysis.

Studying the adsorption profiles of endoglucanase and exoglucanase on cellulose will result in a better understanding of cellulase action and may foster the development of better cellulase mixtures. This paper reports the results of a study of the adsorption profiles of endoglucanase and exoglucanase on cotton fibers.

# Preparations of the Nanostructured TiO<sub>2</sub>/Ethylent Glycol (EG) Colloid Solutions as a Precusor for the In Situ Polymerization of PET

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This paper discusses the impact of milled conditions on the particle sizes und their distribution in the preparation of nanostructured  ${\rm TiO_2/EG}$  colloid solution, and the resulted photocatalyst effect is also investigated.

The most effective process for making polymer containing nanostructured particles is *in situ dispersion polymerization*. The merits of this process are in one hand, the nanostructured particles are not coagulated in sol-gel solution; on the other hand, the particles can be dispersed evenly within the polymer. In the present paper, we used 300 nm TiO<sub>2</sub> powder as raw material, milled and dispersed in EG in the ball milling machine, to mill into nano size from 30 to 100 nm. The used TiO<sub>2</sub> powder was a modified type coated with Ti, and the phase was anatase especially for PET polymerization. The concentrations in Wt% were various with 3, 5, 7, 9, 20 and 30 %, respectively.

In the previous research works, the optimal movement trace of milled balls "free falling" was found. Under that situation, in a certain revolution speed, the balls fall and reach their maximal impact forces that act on the milled TiO<sub>2</sub> material. The falling and tumbling balls crash and peel them into small pieces. The milling process was processed in a temperature-controlled container in 5 °C. In this process, we didn't add any disperse agent in the colloid solution for preventing any effects from polymerization in the coming step. The colloid solution was controlled in an acid base with pH values of 6, which results in a well dispersion situation in solution.

We discuss the impact of milling conditions (milling time 1, 3, 6, 9, 12 hr; ball diameters 5, 2, 0.5 mm; ball weight 200, 400, 800, 1000, 1200, 1400, 2000 g) on the particle sizes and their distributions. After milling, the solutions were diluted and tested with Brookhaven Instruments-90 for particle sizes. The tests were measured in two time-intervals of 5 and 10 min, respectively. In addition, the particle sizes were also proved with TEM for comparisons. The UV-visible Recording Spectrophotometer UV-2100 was used to detect the UV-light absorbency of light intensity, wavelength and transparency. Besides, the PH-value and Zeta-potential of colloid solutions were also examined.

In a constant ball weight of 1200 g, solid content of  $TiO_2 9$  %, ball diameter of 2 mm, the particle size of  $TiO_2$  is decreased along with milling time, reaches the optimal value (6 hr in this study), and increased afterward. In the beginning, along with milling time, the particle size is decreased because of the particle cracking due to the impact forces; however decreased after the optimal milling time and coagulate each other due to the increasing heat energy. This result is independent of measured time-intervals. The Zeta-potential also shows a similar trend to prove these phenomena.

In a constant milling time of 6 hr, TiO<sub>2</sub> solid content of 9 %, ball diameter of 2 mm, the particle size of TiO<sub>2</sub> is decreased along with ball weight, reaches the maximal value (1200 g in this case), and increased afterward. In the beginning, the crash efficiency is increased with mass (ball weight); however, the effective contact areas of balls are no long increase when the ball weight reaches a maximal volume. The increasing ball weight increases merely increase the impact on the container' wall that results in an increment of heat and accelerates particle coagulation.

In a constant milling time of 6 hr, ball weight of 1200 g, ball diameter of 2 mm, the particle size of TiO<sub>2</sub> is decreased along with solid content of TiO<sub>2</sub>, reaches the optimal value (9 % in this case), and increased afterward. This reveals a limitation of action of milling. The increment of solid content results in an ineffective of milling action.

In a constant milling time of 6 hr, ball weight of 1200 g, TiO<sub>2</sub> solid content of 9 %, the particle size of TiO<sub>2</sub> is decreased along with ball diameter. The small ball diameter increases the contact areas of particle and balls. The smallest ball diameter used in this study was 0.5 mm and the mixture of ball diameter or finer ball diameter is not involved in this case.

The UV-light absorbency shows an obvious relationship with particle size. The smaller the  $TiO_2$  particle size is, the higher is the UV-light absorbency, e.g. the UV-light absorbency is 87.9 % corresponding to a  $TiO_2$  particle size of 38.7 nm, where the UV-light absorbency is 29.8 % corresponding to a  $TiO_2$  particle size of 184.5 nm.

We didn't find any concrete conclusions between the PH values of milled particles and milling conditions. Consequently, for the milling conditions, we believe that the most important factor for particle size is milling time, follows ball weight, ball diameter and solid content in the condition of an optimal ball movement.

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- ITMF (Swiss)
- International Textile Academia(Swiss)

#### **PUBLICATIONS**

Journal Papers: 46 Conference Papers: 98 Technical Articles: 49

Books: 4

#### **PATENTS**: 2

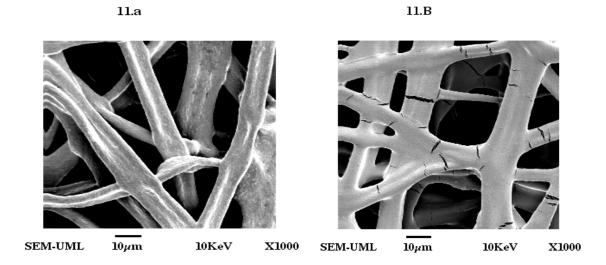
# Structure and Transport Properties in Electrospun Butyl Rubber Membranes Nantiya Viriyabanthorn, Jamila Shawon, Joey L. Mead, and Ross G. Stacer Department of Plastics Engineering, University of Massachusetts Lowell

Development of breathable elastomeric membranes based upon commercial butyl rubber solution coating compounds in reported. These semi-permeable membranes are produced by electrospinning the compounded butyl rubber stock from solution under appropriate conditions. They are designed to be selectively permeable, allowing the transport of moisture vapor, while resisting the passage of liquid water as shown in Figures 1 and previously reported.<sup>1</sup>

Electrospinning of thermoset elastomers is a new method for producing nonwoven materials for applications requiring high elongation (stretch) and porosity. This research studied the effect of electrospinning parameters such as solution viscosity and voltage on the morphology of uncured materials. A reduction in the solution viscosity resulted in the expected reduction in fiber size. Increasing the voltage, while maintaining the same distance between the pipette and target resulted in less distinct fibers with a flattened appearance. Curing of the electrospun mat results in a change in the morphology of the fibers, appearing to increase the connectivity between fibers as shown in Figure 2. Average mechanical properties for the electrospun mats are shown in Table 1.



Figure 1. Selectively Permeable Butyl Rubber Membranes.



A. Uncrosslinked material

B. Crosslinked/cured material

Figure 2. The effect of curing on fiber morphology.

Table I. Mechanical Properties of Electrospun Butyl Rubber

Material	Max. Stress	Ultimate	100 %	300% Modulus
	(Mpa)	Elongation (%)	Modulus (Mpa)	(Mpa)
Butyl Rubber	2.8	1300	0.6	0.275

(1) N. Viriyabanthorn, M. Kam, J. Mead, and R. Stacer, "Barrier Properties of Butyl Rubber Membranes Based on Electrospinning Process", Poster presented at a meeting of the Rubber Division, American Chemical Society, Cleveland, Ohio, October, 2001.

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# Characterization of Beaded PU Nanofibers Deposited on Electrospun PET Nonwovens

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#### 1. Introduction

Electrospinning is a process by which polymer nanofibers can be produced using an electrostatically driven jet of polymer solution. Electrospun fibers are collected in the form of non-woven mats, which are of interest for a variety of applications such as semi-permeable membranes, filters, composite reinforcement, and scaffold used in tissue engineering[1,2]. Electrospun fibers often have beads in regular arrays[3]. The viscosity of the solution, voltage, and the surface tension of the solution are important role in influencing the formation of the beaded fibers. Effects of beaded fiber upon extraordinary behavior of the property might be significant. Changes in structure are also evident in scanning electron microscope(SEM) images of the bead/fiber rearrangement as the spinning conditions are exchanged. Electrospun beaded fiber coatings with polyurethane(PU), were applied directly to polyester nanofiber membrane. The wetting ability and air flow properties correlate with the number and arrangement of beads. Electrospun beaded fibers produce an exceptionally complicated multifunctional membrane for protective clothing applications, which have different wetting ability, elasticity and filtration efficiency.

### 2. Experimental

The solution were prepared at room temperature, and gently stirred to speed dissolution. Prepared solution was held in a syringe, which had an orifice of 0.5 mm diameter in the end. The syringe was connected to a high-voltage supply, which is capable of generating positive DC voltages up to 50 kV. The tip of the syringe can be placed vertically over the cathode or at any other convenient angle to it. This experimental consists of two procedure. At first, the electrospun polyester nonwovens were collected on aluminum foil. To make a similar structure of leaf of lotus flower, electrospun PET nonwovens were deposited by electrospun beaded PU nanofibers. The fiber diameter and polymer morphology of the electrospun PU beaded fibers were determined using SEM. A small section of the non-woven mat was placed on the SEM sample holder and sputter-coated with gold. A lotus flower, showed good waterproof efficiency, was also observed to be compared with the structure of beaded fibers. The gas permeation analyzer(GPA) was used to measure permeability of samples.

#### 3. Results and discussion

Fig. 1 shows the representative images of beaded PU nanofibers and PET/beaded PU complex. Also, the leaf of lotus flower was investigated. The surface tension and viscoelastic properties of the polymer solution are the key parameters in the eletrospinning process. Beads and beaded fibers are less likely to be formed for the more viscous solutions. By changing parameters of electrospinning, we controlled the beads size and number of beads. The high waterproof efficiency of lotus flower, as was shown in Fig. 1, is the result of characteristic structure on front and back of leaf. From this fact, the membrane composed of PET/ beaded PU complex, with the same structures of lotus flower, was prepared by electrospun. Electrospun PU beads were applied directly on the surface of electrospun PET nonwovens. The PET/beaded PU complex showed a good waterproof and gas permeability. These results demonstrate that this complex may be useful for protective clothing systems and filter used in humid surroundings.

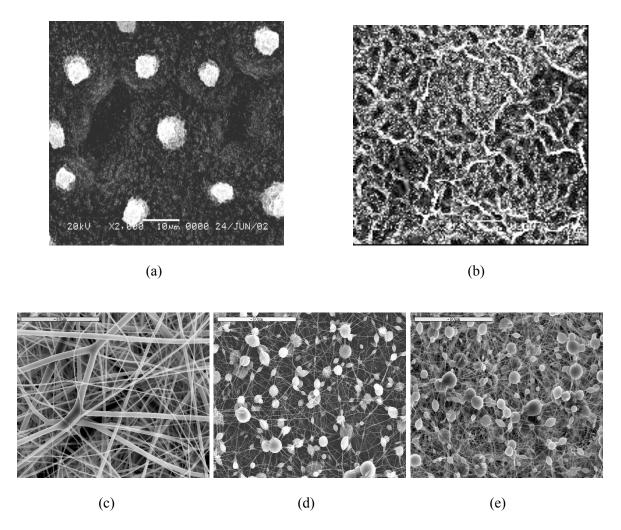


Fig. 1 SEM images of a) front side of leaf of lotus flower, b) back side of leaf of lotus flower, c) electrospun PET nonwovens, d) electrospun beaded PU nanofibers and e) PET/beaded PU complex, respectively.

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# A Study on Electrospun Poly(vinly acetate) Nonwovens Hak Yong Kim<sup>1</sup>, Keun Hyung Lee<sup>2</sup>, Kyung Ju Choi<sup>3</sup>, Bong Souk Lee<sup>1</sup>, Douk Rae Lee<sup>1</sup>

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#### 1. Introduction

Electrospinning is an unique method that produces fibers with diameter ranging from nano to a few microns using electrically driven jet of polymer solution and melt [1-2]. This method was introduced to Formhals in 1934 [3]. Nonwovens composed of electrospun fibers have a large surface area per unit mass and small pore size, so are of interest in a wide variety of applications such as filtration, reinforcing fibers in composite materials, biomedical device [4, 5]. The morphology of the electrospun fibers depends on various processing parameters such as solution properties, controlled variables and ambient [6]. Therefore, optimal nanofibers can be prepared as controlling these parameters.

#### 2. Experimental

#### 2.1. Materials and Solution Preparation

Poly (vinyl acetate) (PVAc) of molecular weights 140,000 (B-25) and 500,000 (B-100) were purchased from McGean (USA). These chemicals were used without further purification. The four solutions were prepared. Sample A and B were prepared by dissolving B-25 and B-100 in acetone, respectively and having from 5 to 15 wt% in solution. Sample C and D were prepared by dissolving B-25 and B-100 in the mixture of methanol and ethanol ratios of 100/0, 80/20 and 60/40, respectively.

#### 2.2. Measurements

Both surface tension and viscosity of polymer solutions were determined by the Wilhelmy plate method using tensiometer (K10ST, Krűss Co., Germany) and Brookfield digital viscometer (Model DV III, USA) at 25°C, respectively. The morphology of the electrospun fibers was examined with scanning electron microscopy (SEM, GSM-5900, Jeol. Co., Japan). The fiber diameter and its distribution were obtained by using image analyzer (Image-proplus, Media

Cybernetics Co., USA) from the SEM photographs. Gas permeability was determined by gas permeation analyzer (GPA-2001, B. S. Chem. Co. Ltd, Republic of Korea).

#### 3. Results and Discussion

Two different molecular weight of PVAc dissolved in two types of solvent system were electrospun to prepare fibers. As the results, the molecular weight of the polymer is to be very important in preparing nanofibers. PVAc nonwovens composed of nano-microns fibers have successfully prepared by controlling various parameters (Fig. 1). As shown these SEM image, fiber diameters can be changed as controlling electrospinning parameters. Viscosity of the solution is the key factor determining the electrospun fiber diameters. It can be controlled as changing polymer concentration. Gas permeability of electrospun nonwovens depends on fiber diameters.

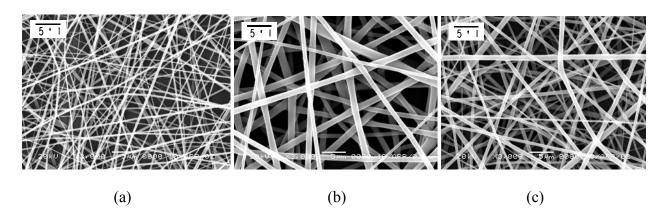


Fig. 1. SEM image of PVAc (B-25) fibers produced by electrospinning solution dissolved in acetone: (a) B-100, 7 wt% (b) B-100, 15 wt%, (c) B-25, 15 wt%.

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# **Electrospun Hybrid Nanofibers of Alumina-Boria-Silica**

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#### 1. Introduction

Alumina fiber is an important engineering material as used in various applications, which include high-temperature insulation material, catalyst supports in high-temperature reactions, fire protection, and as reinforcement for resins, metals and ceramic [1]. Alumina hybrid materials, Al<sub>2</sub>O<sub>3</sub>-B<sub>2</sub>O<sub>3</sub> –SiO<sub>2</sub> ternary systems, have also aroused great interest. These materials exhibit some attractive properties like refractoriness, low thermal expansion and conductivity, good chemical stability, and excellent mechanical properties at high temperatures [2]. In the present work, a new method to make alumina-boria-silica hybrid nanofibers was introduced.

#### 2. Experimental

Both the aluminum acetate stabilized with boric acid and silica sol were added to PVA (poly(vinyl alcohol),10 wt%) solution with vigorously stirred for 24h at room temperature. These mixtures were aged for about 5h at room temperature and a viscous solution of alumina-boria-silica/PVA was prepared. Alumina-boria-silica/PVA hybrid fibers were electrospun using electrical force. The hybrid fibers were treated at 1000 and 1400 °C for 24h under ambient atmosphere.

#### 3. Results and discussion

Element analysis data showed that all the organic materials were removed above 1000 °C. The SEM photographs of alumina-boria-silica hybrid fibers were shown in Fig. 1. As shown in Fig. 1, the fiber diameter was 150-200 nm. Notably, it could be seen that the surface of fibers became rougher for treating higher temperature. FT-IR was shown that the characteristic peaks of PVA disappeared after treating above 1000 °C and some new peaks appeared. The peak at 1000-1020 cm<sup>-1</sup> indicated the Si-O-Si asymmetric stretching vibration. A strong band at 610 cm<sup>-1</sup> was corresponded to the formation of Si-O-Al bond[2]. Peaks which appeared at 1200-1500 cm<sup>-1</sup> may be attributed to the formation of crystalline aluminum-borate [3]. The peak at 560 cm<sup>-1</sup> should be assigned to Al-O stretch [4].

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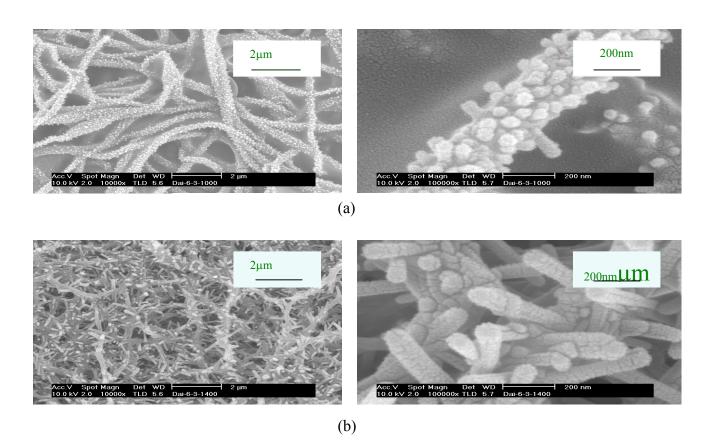


Fig. 1. SEM photographs of alumina-boria-silica hybrid fibers ((a) treatment at 1000 °C, (b) treatment at 1400 °C).

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# Carbon Nanofibers Hybridized with Palladium Nanoparticles Produced by Electrospinning

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Electrospinning is one of the effective methods to produce nano-structures. In the past decade, a series of efforts have been made to produce polymer nanofibers <sup>(1-3)</sup>, nanotubes <sup>(4-5)</sup>, and nanocomposite polymer <sup>(6)</sup> by using electrospinning.

In the recent study, polyacrylonitrile (PAN) nanofibers hybridized with palladium acetate (Pd(Ac)<sub>2</sub>) were produced by using electrospinning of DMF solution of PAN and Pd(Ac)<sub>2</sub>. The hybrid nanofibers were annealed at 1100°C in Ar atmosphere. The observation by transmission electron microscopy (TEM) and electron diffraction shows that the hybrid PAN fibers converted to carbon and Pd-particle hybrid nanofibers (see Fig. 1).

Using the hybrid carbon fibers as the catalyst of carbon nanotube growth and acetylene as an additional carbon source, some short carbon nanotubes were found to grow on the surface of the hybrid carbon fibers at 650-700°C. TEM observation shows in Fig. 2 that the Pd-nanoparticle locates at the tip of the grown nanotube as a catalyst for the formation of carbon nanotube.

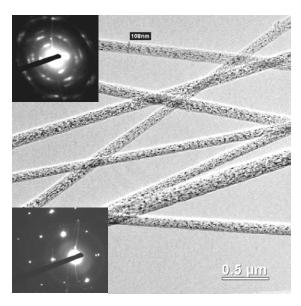


Fig.1. TEM image of carbon nanofibers hybridized with Pd-nanoparticles produced via annealing of PAN and Pd(Ac)<sub>2</sub> hybrid nanofibers at 1100°C in Ar atmosphere. The top insert is the electron diffraction pattern of carbon fiber, and the bottom insert is the electron diffraction pattern of Pd-nanoparticle.

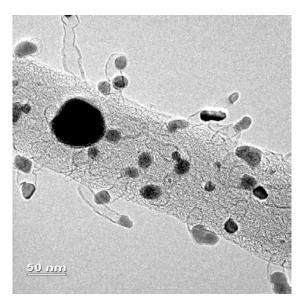


Fig.2. TEM image of carbon nanofibers with carbon nanotubes produced by using chemical vapor deposition (CVD) pyrolysis of acetylene on the carbon/Pd composite fibers at 650-700°C in Ar atmosphere.

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# **Melt Electrospinning of Thermoplastic Polymers**

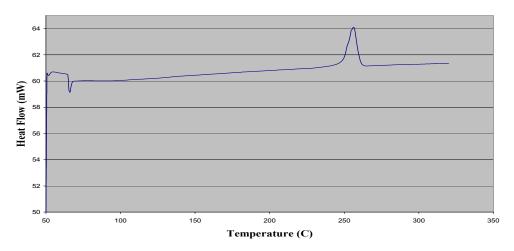
# Jason Lyons, Frank Ko, Amotz Geshury

Drexel University, Fibrous Materials Research Lab

There has been a great deal of research devoted to the electrospinning of nanofibers from solution, yet little research has been devoted to spinning nanofibers from the melt. This work is aimed at developing and optimizing the parameters that are capable of producing continuous submicron to nanosize thermoplastic fibers using the technology of melt electrospinning.

Initially, heating devices were used to obtain a polymer melt capable of being electrospun. Once in the molten state, a variety of spinning angles, spinning distances, and electric fields were used to form fibers. The relatively large fibers produced using this method showed a lack of crystallinity resulting in exceptionally weak fibers over a very large diameter range. The amorphous nature of the fibers is attributed to the fiber quenching as soon as it leaves the spinning chamber.

#### **DSC Heating Commercial Polyester**



#### **DSC Heating E-Spun Polyester**

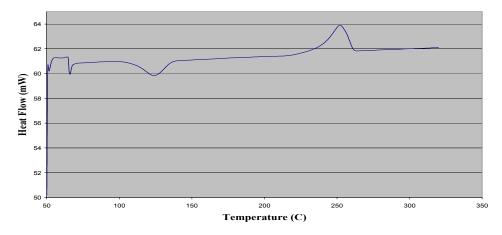


Figure 1: Differential Scanning Calorimetry of Commercial and Electrospun Polyester

The size and shape of the fibers produced varies greatly with the polymer selected, processing temperature, and atmospheric temperature; to name a few. Average sizes from as small as 500 nm to as large as 20 microns were produced. Heating the atmospheric temperature resulted in the finest fibers yet a problem of coagulation of the melt on the collector resulted in the occasional formation of a film, rather then fiber.

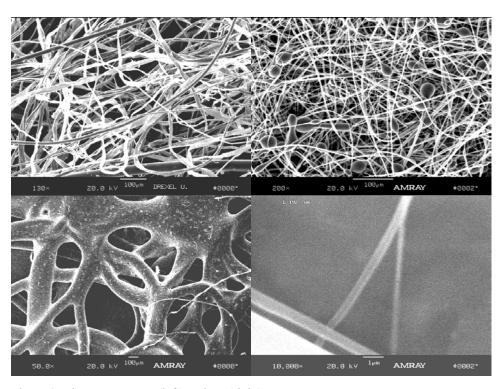


Figure 2: Electrospun PET (left) and PP (right)

Recent work has shown that a conventional polymer extruder can be modified to electrospin micron size fibers. It is the hope that with a very uniform heating chamber and controlled spinning atmosphere; very uniform and crystalline submicron to nanosize fibers can be produced. If this methodology of producing nanofibers is successful, the advantages are numerous. Fibers on the nanoscale that can be used in many industries will be created using a spinning technique that results in a 100% yield and is not only economically friendly, but environmentally friendly.

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Jason is a third year graduate student at Drexel University pursuing his doctorate degree in Materials Engineering. He works in the area of polymeric materials and concentrates on the production of thermoplastic fibers to compliment his bachelor's degree in Textile Engineering. In the past, Jason has done work on yarn, fabric, and composite mechanics and continues his studies in the characterization on melt electrospun thermoplastic textiles.

# Hand Value by Wet Cleaning of Wool Fabric

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#### Introduction

The dry cleaning was necessary and indispensable in the cleaning industry. As for a nonflammable solvent, tetrachloroethylene and the Tori fluorination trichloroethane (CFC113) were used. However, CFC113 used as a mild solvent became a manufacturing prohibition because of the material, which destroyed the ozonosphere. Tetrachloroethylene caused the pollution of the soil and underground water, and, in addition, the doubt of causing carcinogenic and the affection of the liver came out from this, too. Only the petroleum solvent is being used now. It is forecast to need washing the water system (wet cleaning) now. Advantage of a wet cleaning is harmless in the human body and the environment in the first. Next, the water washing often removes the dirt of the water-soluble. However, clothes are set to finish up the general view beautifully because of heat and steam. However, because the water molecule is very small compared with the structural unit of wool, it becomes a factor to destroy the hydrogen bond of the alpha helix structure. In addition, because making to the felt advances when the mechanical power is added to wool, the hand of the fabric is decreased. In this research, a confirmation of the hand change of the fabric by a wet cleaning and what kind of mechanical factor influences the hand value change is studied.

## Experiment

## Experiment sample

For this experiment, 100% wool autumn/winter weight fabric was judged to be ideal. Fabric capable of being washed in water using the BAP process was utilized.

## Washing machine

The compression method washing machine ("Oshi no Itute") was developed for industrial wet cleaning in Japan. This machine places laundry between two plates. The plates press the fabric together while moving in a figure-8 pattern. Since fabric damage is minimized and pleats and creases are mostly unaffected, this technique is optimal for wet cleaning. The "Electrolux" washing machine is a drum type developed for wet cleaning of delicate clothing featuring a reduced diameter drum to avoid the 'beating' effect of conventional drum washing machines, inducing more of a 'pushing' wash effect.

## Washing method

20cm squares of suit fabric were used. In order to provide a baseline, MA cloth was loaded at the same time. After the sample had been washed, press finishing using the same conditions was used. Samples were measured using a KES-AUTO automatic system after 0, 3, 5, 10 and 15 washings. Measurement results were analyzed using a PC.

#### Result and discussion

## Measurement result of MA examination cloth

The MA value of the compression washer was 14.7, compared to 26 for the Electrolux washer. Thus, in terms of mechanical load on the laundry, the compression washer has the advantage.

# Hand value of wool cloth (unprocessed)

Figure 1 shows the hand value comparison for the two wet cleaning methods. THV of the compression washed samples is increased to the 0 to  $3^{rd}$  power, decreasing as washing frequency increases. THV of the Electrolux samples decreased as cleaning frequency increased. Thickness  $T_0$  and compression energy WC of the compression washed samples increased with washing frequency. Flexural rigidity B decreased rapidly with the first washing and decreased gradually wish subsequent washings.  $T_0$ , WC and MIU (surface coefficient of friction) were also changed for the Electrolux samples. Because mechanical power loaded to the sample was greater than that of compression washing, the variation for  $T_0$  and WC was approximately 1.5x. In addition, MIU increased significantly.

# Hand value of wool cloth (BAP processing)

BAP processed wool cloth showed no change in THV and TAV, even with increased washing frequency. However, this cloth did show a thickness decrease as washing frequency increased. No hand decrease due to water washing was observed; therefore, scale removal and/or resin coating for the wool is unnecessary. Such processing reduces the functionality of the wool fabric; however, if the finished product is intended for home washing (and concomitant higher forces loaded to the fabric), this processing may be necessary.

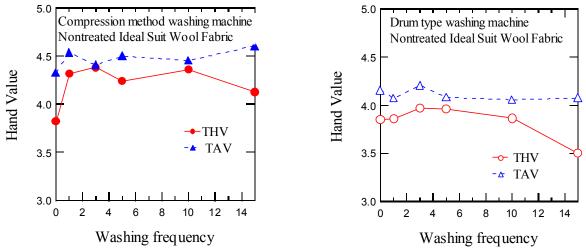


Fig. 1 Effect of the washing frequency to THV and TAV

# Absorption of Fluids by Fibrous Substrates and Nanofiber Webs Konstantin G. Kornev<sup>1</sup>, Alexander V. Bazilevsky<sup>2</sup>, Aleksey N. Rozhkov<sup>2</sup>, and Alexander V. Neimark<sup>1</sup>

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When a fibrous substrate is set in contact with a wetting fluid, the fluid spontaneously wets solid surfaces and penetrates inside pores. A better understanding of spontaneous spreading and penetration is required for improved characterization of fabrics, nonwovens, nanofibrous webs, and the like. This phenomenon involves different characteristic times and scales. We study physical mechanisms of the fluid absorption focusing on the initial stage of uptake. This regime is characterized by surprisingly large Reynolds numbers, which may exceed 100! Thus, in contrast to the Lucas-Washburn kinetics associated with a slow creep of a liquid column, the viscous forces seem to be negligible compared to the inertial and capillary forces. We have developed an optical electronic measuring system to monitor the spontaneous penetration of droplets in capillaries and porous substrates with a millisecond resolution. The time of absorption of a droplet of a given volume characterizes the material absorbency. We experimented with different fabrics, papers and electrospun nonwovens. An analysis of the rate of absorption of viscous, colloidal, and viscoelastic fluids (such as polyacrylamide (PAM), polyethyleneoxide (PEO) and blood and mucus substitutes) was performed to show that the fluid rheology affects the uptake rate significantly. Measured on the extensional viscometer, the PAM and PEO solutions manifest a typical viscoelastic response thus pointing out that the elastic response of complex fluids is crucial. Dissolved in water, a small amount of PAM (that does not change the shear viscosity of the solution and the surface tension) gives rise to the sixfold reduction of the rate of absorbency. Experimental observations on capillaries are in agreement with the model, which attributes the velocity reduction to the Weissenberg effect of the normal stress difference present in flowing complex fluids. The model generalizes the Lucas-Washburn equation to account for the inertia, and the viscoelastic friction of meniscus. Unlike the Poiseuilian friction force, which is proportional to the product of the length of liquid column and meniscus velocity, the viscoelastic friction force is independent of the length of liquid column. It is proportional to the meniscus velocity squared. That is, the fluid elasticity does affect the flow while the Poiseuilian friction force does not.

# Numerical Calculation of High Speed Traversing Motion in Weaving and Winding *Manuel Spoerri*

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Textile machines working at a very high speed are the state of art in textile manufacturing. In many textile processes, in particular weaving and winding, mechanical elements move at high speeds with reversal of direction at a given position. These elements must move with very high acceleration and deceleration rates, examples are the beat-up of a loom or the traverse motion of a winder. Therefore, these mechanical structures are highly stressed.

In the first part of this paper, it will be shown what physical conditions are extant in a typical application and how these conditions limit the application itself. There are currently mechanical limitations to the transmission of the instantaneous energy flows, as for instance the material strength, which occur during changes in the direction of motion. Investigations of different physical principals have been done and discussed to realize such acceleration rates, supporting or substituting a conventional power transmission (e.g. gear box): magnetic drive, mechanical and gas spring. Calculation has shown, that the most performing and simple element is the gas spring reflector. Therefore, it has been chosen for further analysis.

In the second part of this paper a concept is presented of a new method of changing the direction of motion of a linearly moving mechanical component. With this method, both mechanical complexity and stress, and energy consumption will be minimized: the pneumatic motion reflector.

First a physical model of the system has been developed and realized in a computer program to simulate the effects (e.g. pressure, temperature) during the reflection. Many physical effects had to be taken into account, establishing the model, such as leakage, mechanical friction and so forth. The purpose of this work was to predict the energetic degree of efficiency: how much of the initial energy is remaining in the system after having reversed the direction of motion, without feeding in energy.

Afterwards a basic experimental apparatus has been constructed to verify the model. A mass is accelerated from zero to a certain speed and brought into the motion reflector, where its motion is reversed. After this cycle, the mass is decelerated to zero again and brought into the initial state. Two sensors are attached to the construction: the first sensor measures the velocity of the mass before and after the reflection, the second detects the pressure of the gas in the reflector.

The final chapter examines the possibilities of incorporating this new concept into future textile machines.

# **Electrospinning of Biopolymers**

# Chen-Ming Hsu, Jing Tao and Satya Shivkumar

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Polymers such as poly(caprolactone), PCL, and poly(vinyl alocohol), PVA, are being used in many biomedical applications including tissue engineering and drug delivery. PCL is a semicrystalline aliphatic polyester that is rubbery at room temperature and has a much smaller *in vivo* degradation rate than PGA or PLA. It can also exhibit a good permeability to a wide range of antibiotics [1]. PVA is hydrophilic and a water soluble polymer that is used extensively in wound care, drug delivery and in ophthalmic applications. PVA can be crosslinked by chemical or radiational methods to make a hydrogel with adequate mechanical strength [2]. The purpose of this work was to use electrospinning to produce nano-scale powders, fibers and porous structures from these polymers. The feasibility of incorporating drugs and/or other biomolecules for controlled release during resorption of the polymer has also been examined. PCL with a MW = 40,000 g/mol was dissolved in chloroform to produce solutions with concentrations ranging from 2 to 20%. PVA with a MW between 10,000 to 30,000 was dissolved in water at 90°C to produce aqueous solutions with concentration ranging from 20 to 30 wt%. The resulting solutions were electrospun at voltages between 20 and 40 kv. In some experiments, acetominaphen or other biomolecules were added to the solution to examine the feasibility of controlled drug release. Typical examples of the fibers produced after electrospinning are shown in Fig. 1.

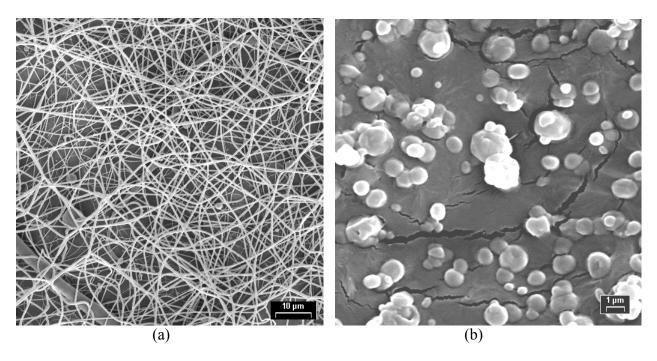


Fig. 1 Examples of structures produced with PCL for 2 different concentrations. a) 8 wt% b) 3 wt%.

The fiber diameter (typically between 400 to 800 nm) and the resulting structure after the deposition of the fiber on the substrate, could be varied by controlling the voltage and concentration. The resulting structure had a broad distribution of interconnected pores and can be used as a scaffold for tissue engineering. At low voltages, powders with diameters between 0.1 and 2 µm could be produced. The effects of MW, crosslinking agents and other additives on the structure of the electrospun product are currently being examined through the use DSC, TGA and IR spectroscopy. The kinetics degradation in physiological solutions and the associated release rates for biomolecules are currently being measured. The results indicate that electrospinning can be used to produce unique structures from these biopolymers for various physiological applications.

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# Author Biography

Chen-Ming Hsu is a graduate student pursuing his M.S. degree in Materials Science and Engineering at Worcester Polytechnic Institute, Worcester, MA. He got his B.S. degree in Chemical Engineering from Yuan-Ze University, Taiwan in 1996. His research interests include polymer processing and biomaterials.

# Structural and Conformational Characterization of Poly(ethylene 2,6 naphthalate) by Infrared Spectroscopy

#### Nad Vasanthan

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Poly (ethylene 2, 6 naphthalate) (PEN) is a crystallizable polyester which can be used in higher temperature applications compared to PET. The naphthalene ring provides greater rigidity to the polymer backbone than the benzene ring in PET, elevating the glass transition temperature and melting point and enhancing the mechanical properties such as tensile modulus and creep resistance. Crystallization of PEN has been investigated by Zachman et al who reported that it can crystallize into two different crystal modifications. The  $\alpha$  crystal form can be obtained by annealing amorphous PEN in the solid state and the  $\beta$  crystal form can be obtained by crystallizing directly from the melt. The unit cell of the  $\alpha$  crystal form, determined by Mencik et al, is triclinic with a=0.651, b = 0.575, c=1.32nm,  $\alpha$ =81.33°,  $\beta$ =144° and  $\gamma$ =100° and a density of 1407 kg/m³. The chains lie parallel to the c-axis and a single chain passes through the unit cell. A triclinic unit cell was also proposed for the  $\beta$  crystal form with a=1.273, b=1.559, c=1.273nm,  $\alpha$ =121.6°,  $\beta$ =95.57°, and  $\gamma$ = 122.52°. Four chains pass through each unit cell of the  $\alpha$  form and, since every naphthalene ring is rotated by 180°C, the chains are not completely extended. The density of the  $\beta$  crystal form was reported as 1439 kg/m³.

X-ray diffraction analysis demonstrated that PEN film annealed from 160° C to 260°C, and crystallizes in the  $\alpha$  crystal form while crystallization of PEN directly from the melt results in the  $\beta$  crystal form when  $T_c > 200$ °C and in the  $\alpha$  form at lower  $T_c < 200$ °C. Infrared spectra taken in the region between 600 to 1800 cm<sup>-1</sup> at room temperature for amorphous PEN and for semicrystalline PEN containing the  $\alpha$  and the  $\beta$  crystal forms are presented in Figure 1a, 1b and 1c, respectively. The changes with crystallization taking place in the spectrum for a particular sample can best be followed if difference spectra are used. Digital subtraction of the amorphous component was carried out from the semicrystalline spectra by observing the disappearance of the amorphous bands at 1453 and 824 cm<sup>-1</sup> to obtain characteristic spectra of each phase.

The amorphous spectrum shows a band at 1714 cm<sup>-1</sup>, associated with the C=O stretching vibration, while it appears at 1709 cm<sup>-1</sup> for the  $\alpha$  crystalline phase and 1707 cm<sup>-1</sup> for the  $\beta$  crystalline phase. The observed band shift for the carbonyl stretching vibration in the crystalline phase is due to differences in the conjugation between the carbonyl group and naphthalene ring. Another important feature is that the amorphous spectrum shows a strong broad band at 824 cm<sup>-1</sup>, assigned previously to CH<sub>2</sub> rocking vibration while three new bands at 839 and 814 cm<sup>-1</sup> appear during the crystallization of  $\alpha$  crystal form and one band appears at 837 cm<sup>-1</sup> for the  $\beta$  crystal form of PEN. The band at 824 cm<sup>-1</sup> can then be attributed to the amorphous phase, the band at 839 and 814 cm<sup>-1</sup> can be attributed to the  $\alpha$  crystalline phase and the band at 837 cm<sup>-1</sup> to the  $\beta$  crystalline phase. It has been suggested that these new bands appearing in the crystalline spectrum arise from inter chain interaction, somewhat similar to crystal field splitting observed in polyethylene.

In order to confirm the assignments of crystalline and amorphous bands, correlation between polymer density and the absorbance of bands at 824 and 814 cm<sup>-1</sup> was carried out. The absorbance ratios of the 814 and 824 cm<sup>-1</sup> bands to the internal reference band at 1602 cm<sup>-1</sup>, attributed to naphthalene ring vibration, were then plotted against the measured density and fitted by linear regression, as shown in Figure 2. A linear dependence of the normalized absorbance with respect to density is observed. The extinction of the band at 814 cm<sup>-1</sup> gave a density of 1324 kg/m³, in close agreement with the reported amorphous density of 1325 kg/m³, confirming its attribution to the crystalline phase. Extrapolation of the band at 824 cm<sup>-1</sup> to zero absorbance provided a density of 1404 kg/m³ and confirms its amorphous phase origin. However, when the band at 839 cm<sup>-1</sup> was plotted against density (not shown) extinction of this plot provided a density of 1300 kg/m³, much lower than the reported amorphous density, indicating that the 839 cm<sup>-1</sup> band cannot be fully attributed to the crystalline phase.

The bands at 1004, 1332 and 814 cm<sup>-1</sup> were attributed to all trans conformation in the  $\alpha$  crystalline phase and the bands at 975 and 1348 cm<sup>-1</sup> were attributed to the  $\beta$  crystalline phase. Our infrared spectroscopic investigation suggests that the  $\alpha$  crystal form adopts an all trans conformation while the  $\beta$  crystal form adopts a conformation with appreciable gauche character. Conformational changes occurring in PEN are not only due to the rotation of the ethylene glycol unit but also arise from the rotation of the naphthalene ring. This is different from PET, where conformational changes have been observed to occur only in the ethylene glycol unit.

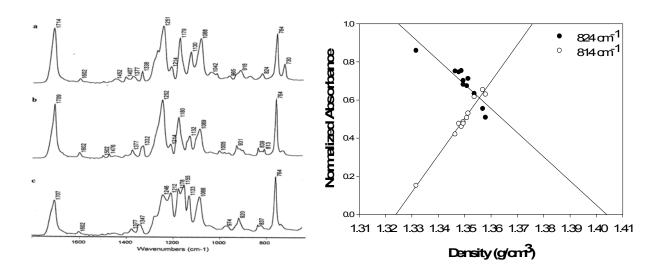


Figure 1 Figure 2

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# **Biographical Information**

Dr. Nadarajah Vasanthan, Senior Scientist, joined at TRI/Princeton in 1997 after 4 years of polymer research at North Carolina State University as a research associate and AlliedSignal as a research scientist. He earned a Ph.D. degree in Polymer Physics from City University of New York and B.Sc in chemistry from University of Jaffna, Sri Lanka. His research interests include crystallization and microstructure characterization of fiber forming semicrystalline polymers, biodegradable polymers and spectroscopy. Dr. Nad's research accomplishments are reflected in his more than thirty research publications in reputed polymer journals.

# **Elastomeric Selectively Permeable Membranes for Chemical and Biological Protective Clothing**

# Quoc Truong<sup>1</sup>, Shantha Sarangapani<sup>2</sup>

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Fabrics containing elastomeric barriers such as butyl rubber provide excellent protection from chemical and biological (CB) warfare agents, but wearers can use the clothing only intermittently due to rapid onset of heat stress, motion restriction, and weight. Air permeable activated carbon-based fabric systems such as the US Army Battle-Dress Overgarment (BDO) [MIL-S-43926], the Joint Service Lightweight Integrated Suit Technology (JSLIST) overgarment (NSN 8415-01-444-1238), and other uniforms in use by military forces worldwide provide the individual soldier chemical warfare (CW) agent vapor protection for an extended period of use. However, these fabric systems are also heavy, bulky, and subject the soldiers to heat stress under high workload and in battlefield conditions. NSC has developed new selectively permeable membranes (SPM) in collaboration with the membrane industries such as W.L. Gore & Associates, Inc, Acordis Research GmbH, and W.R. Grace & Co., and to allow selective diffusion of water molecules for evaporative cooling while providing protection against CB agent threats with minimum heat stress, weight, and bulkiness (Figure 1).

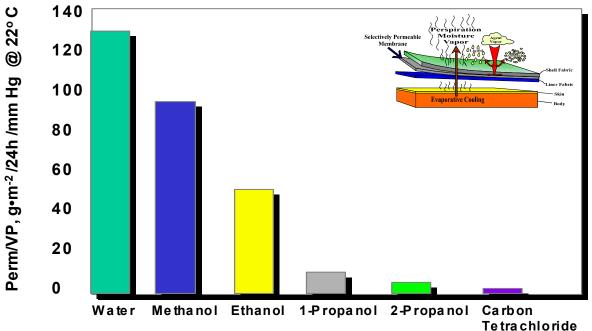


Figure 1. Permeation/Saturated Vapor Pressure of a Typical Selectively Permeable Material.

SPM technology developments have their origins from the membrane industry for carrying out gas separations, in the purification of water by reverse osmosis, and in medical applications.<sup>3,4</sup> Although, these selectively permeable membranes are proven effective barriers to CB agents (Figure 2), they are non-elastomeric. They cannot be used in making conformable clothing, which are sought for in advanced material development programs such as the Advanced Integrated Warfighter Protection, the Scorpion, and the Objective Force Warrior programs.

Dermal penetration of CW agents is rapid and could be lethal (skin permeability rates for mustard and nerve agents are 2.0 and 0.1 cm/min respectively.) While biological warfare (BW) agents do not penetrate the skin, they could adsorb on to clothing or gloves in high concentrations and will cause extensive environmental bio-contamination. Besides, the entry of infection through cuts, abrasions, and bruises on the skin poses a percutaneous threat as serious as inhalation. E-SPMs provides passive physical barrier to significantly retarding or stopping the diffusion of the CW agents and other solvent molecules. They will also provide reactive barrier properties. In the event some diffusion of CW agents occurs through the surface layers, the polymer matrix provides catalysts that enhance the hydrolysis of the CW agents. E-SPMs also act as active biocidal barriers. The surface of the material provides a "kill zone" for Anthrax and other microorganisms and provides an in-air continuous contact biocidal activity (Figure 2).

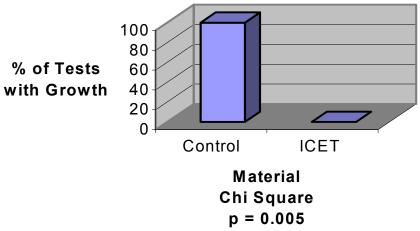


Figure 2. Anthrax Kill-Rate. Performed by ECBC. A. Turesky.

Selected e-SPMs were developed and coated onto fabrics for enhanced durability, and the properties of the lab-size samples were tested for their moisture vapor transport (MVT), barrier (to simulants and surety CW agents), and physical properties. MVT properties were determined using a novel dynamic moisture permeation cell (DMPC) developed at NSC.<sup>5</sup> Barrier properties were determined under various humidity levels using a membrane permeation instrument which NSC developed to study steady state and dynamic organic vapor transport through films, fabrics, and membranes.<sup>6</sup>

In summary, selectively permeable materials (SPMs) have been developed at NSC where they have been proven effective against CB agents while having good moisture transport properties, but their materials are non-elastomeric. NSC and ICET's collaboration resulted in development of an elastomeric SPM (e-SPM). The use of an e-SPM in conformable clothing eliminates the air-boundary layer resistance – effectively maximizing the moisture vapor transmission rate through protective clothing. This paper reports on the significance of the e-SPM concept, material composition, processes, and performance. Discussions will include key properties of these and other materials where their physical properties, barrier properties, and moisture vapor transport properties will be presented. It is anticipated that this novel approach will serve as a viable means of increasing comfort and reducing heat stress in CB clothing, and is the second-generation SPM material.

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#### Biography of Presenting Author

Mr. Truong is a Physical Scientist/Program Manager for research, development, test & evaluation chemical and biological (CB) protection programs at the U.S. Army Soldier and Biological Chemical Command, Natick Soldier Center, Natick, MA since 1984. His recent work involves the development of CB field duty uniforms for the Joint Service, amphibious operation suits for land and air operations, and toxic industrial chemical resistant selectively permeable protective materials for the Memorial Institute for Prevention of Terrorisms, and for other federal and state agencies in preparation for incidents involving weapons of mass destruction. He received his B.S. and M.S. degrees in Plastics Engineering from the University of Massachusetts-Lowell, Lowell, MA, and he is expecting to receive his M.S. degree in Engineering Management from the Western New England College, Springfield, MA in the Fall 2002.

# **Development of Amphibious Operations Suits**

# **Quoc Truong**

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Navy operators desire a low cost, lightweight single garment that can be used for all five phases of a typical operation - insertion, infiltration, actions at the objective, exfiltration, and extraction while remain comfortable in both cold water and warm air environments during their missions (see Figure 1). 1NSC, CSS, Naval Academy, Navy Experimental Diving Unit (NEDU), Navy Textile & Clothing Research



Figure 1. The Amphibian Suit



Facility (NCTRF), Mitsubishi and Malden Mills, and other industry members (as industry partners), have been collaborating in the research and development of the Amphibian Suits with the support from the Naval Science Assistance Program (Task Request R-2-99), and the Naval Fleet Innovation Program. This paper reports on the technical findings on the amphibian suit development based on the use of Mitsubishi's shape memory polymeric (SMP) Diaplex® polyurethane membrane and Malden Mills' Polartec® Regulator fleece fabric.

Elastomeric suits such as the Henderson (Millville, NJ) Premium Step-In neoprene wetsuits (Model # N770F-44) provide excellent thermal comfort for the users in the cold water; however, the wearers in wetsuit would soon be succumb to heat stress during extended operations on land. The dry suits such as the Kokatat (Arcata, CA)'s Gore-Tex® Meridian Dry suit) are waterproof; however, the users would not be comfortable in cold water environment (55-65°F) and warm air environment (65-85°F). The amphibian suit

is constructed from an elastomeric shape memory polymer (SMP)<sup>1</sup> that is waterproof, moisture vapor permeable (WP/MVP) membrane and a specially designed commercial fleece liner<sup>2</sup>. The SMP WP/MVP membrane possesses a unique, pre-engineered microstructure that allows vapors to pass generously when its pre-engineered glass transition temperature (T<sub>q</sub>) approaching that of the environmental temperature, but prevent liquid penetration due to its nonporous material structure (see Figure 2). SMP can be applied to most any backing material. The SMP membrane is laminated to a shell material for durability and appearance. The amphibian suit fabric system is combined with a specially designed fleece material for thermal insulation, which has little interference with the water vapor transport properties of the SMP membrane and shell fabric layer. Thus, minimize the impact on the evaporative cooling of the wearer's body (see Figure 3). These are the characteristics necessary in an

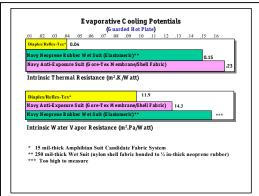


Figure 2. Water Vapor Resistance of the Amphibian Suit vs. Wet Suit, and Dry Suit Fabrics

Amphibian Suit where the suit is a thin, lightweight, self-regulated, waterproof, moisture vapor permeable garment that is suitable for land and water dual-use operations. Such a garment keeps the operator warm and dry by keeping water out, while minimizing body heat loss during swimming operations, but also helps to keep him/her cool during surface operations by letting perspiration pass through the fabric.

In summary, selected SMP MVP/WP fabric systems were developed with favorable amphibious operational characteristics.3 These fabric systems were tested for their performance as compared to the control commercial and current military fabrics. Testing include swatch, mannequin system (garment) testing, and manned testing in climatically controlled chambers and hydroenvironmental (immersion) tanks to measure the physical properties, moisture vapor transport properties, thermal insulative properties, and liquid penetration resistance. The ability of the SMP membrane's elastic memory that responds to the heat and changes its shape to close and to open its molecular structure was investigated, and its interesting

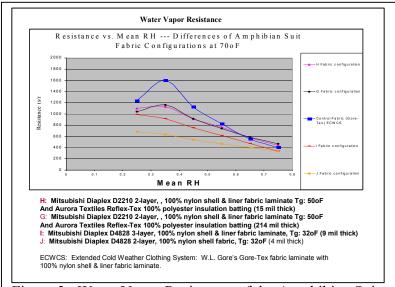


Figure 3. Water Vapor Resistance of the Amphibian Suit Fabric Configurations vs. Control Fabric.

SMP material behavior were experimentally observed. Its elastomeric properties were compared to the Navy/Air Force's current non-stretchable Anti-Exposure Gore-Tex® II Suit (AES).<sup>4</sup> Appropriate 4-way stretchable shell and liner fabrics were also selected for use as integral protective components of the Amphibian Suit. These comparative test results will be discussed along a brief discussion of NSC's current efforts and future plans.

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#### Biography of Presenting Author

Mr. Truong is a Physical Scientist/Program Manager for research, development, test & evaluation chemical and biological (CB) protection programs at the U.S. Army Soldier and Biological Chemical Command, Natick Soldier Center, Natick, MA since 1984. His recent work involves the development of CB field duty uniforms for the Joint Service, amphibious operation suits for land and air operations, and toxic industrial chemical resistant selectively permeable protective materials for the Memorial Institute for Prevention of Terrorisms, and for other federal and state agencies in preparation for incidents involving weapons of mass destruction. He received his B.S. and M.S. degrees in Plastics Engineering from the University of Massachusetts-Lowell, Lowell, MA, and he is expecting to receive his M.S. degree in Engineering Management from the Western New England College, Springfield, MA in the Fall 2002.

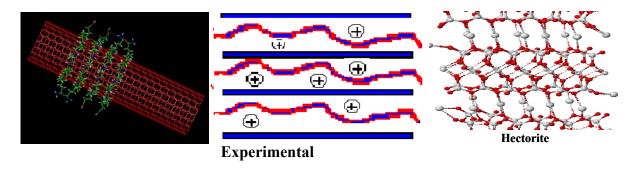
# Electrospun Polymer and Polymer/Clay Carbon Nanotube Composite Fibers Kenneth J. Balkus, Jr., Sudha Madhugri, Raluca Matea, John P. Ferraris, Alan Dalton and Arnvar Zhakidov

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#### Introduction

In spite of the developments in carbon nanotube (CNT) research, there is still a need for improved methods of processing in an easy and practical way. Various polymers have shown potential in threading in or wrapping around the surface of CNTs and acting as non-covalent nanospacers to form stable composites. Here we report a method of producing high surface area polymer-nanotube composite fibers, which is potentially a means to disperse and debundle the CNTs as well as deposit them onto desired substrates.

We have explored the application of electrospinning to process various nanoporous metal oxides and polymer composites. For example we have shown that molecular sieve fibers and polymer composites can be electrospun on to various substrates. We have also shown that nanofibers of PEO and Hectorite clay can be electrostatically deposited. The charged clay appears to facilitate web or paper formation. This technique has now been extended to produce polyethylene oxide (PEO) and polyethyleneimine (PEI)/carbon nanotube (CNT) as well as PEI or PEO/CNT/Hectorite clay composites. The idea is that PEO and PEI polymers with donor sites will wrap themselves around the carbon nanotubes as they are spun into fibers. The clay adds charge and mechanical integrity to the fibers.

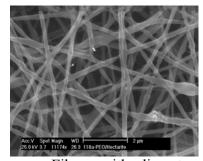


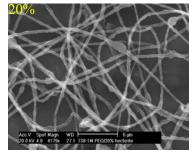
Different molecular weights of PEO and PEI were used to prepare the PEO or PEI/CNT (Hipco single walled nanotubes) (SWNTs) composites. CNTs phase separated from PEO with high molecular weights such as five million. In contrast PEO with lower molecular weights such as 200,000 and 1,000,000 dispersed the CNTs well and kept them in suspension for a few days. A PEO (1 million MW)/10% Hectorite composite was selected to prepare PEO/hectorite/CNTs composite. The carbon nanotubes were present at ~1.5 % by weight in the composites. The method of precursor gel formation included dissolving PEO in deionized water followed by the addition of a definite amount of nanotubes (1.5 wt% in this case) to it with stirring for 24-48 hours. In addition to stirring at room temperature, the dispersions were was bath sonicated for 30 minutes 2-3 times to obtain a uniform dispersion of nanotubes. Resulting solution of pure PEO, PEI or PEO(PEI)/CNTs composite was then placed in a 3 mL plastic syringe barrel, which was connected to a nitrogen driven fluid dispension system (EFD 1500 XL). A positive pressure of nitrogen (7 psi) was applied to maintain a droplet of the viscous gel at the tip of the needle. An

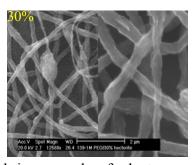
electrode was attached to the needle (22 guage, 1.5" long with a flat tip) at the end of the syringe barrel and the other to the target. A DC voltage of 20 kV was applied to the solution (Soresnsen H. V. Supply (model # 1020-30) which initiates a jet. The composite fibers resulting from the splaying of the viscous gel (~900cp) were collected onto an aluminum foil target positioned 23 cm from the needle tip.

#### **Results and Discussion**

The versatile polymers PEO and PEI, were composited with the layered clay Hectorite, by intercalating the polymer between the layers. The composite samples with 5, 10, 20, and 30% clay were electrospun. The SEM images of a mesh of electrospun PEO fibers (1 million MW) deposited for ~5 min show that the average diameter of the fibers is about 200 nm. PEO fibers containing 10, 20 and 30% w/w clay are shown below. The powder x-ray diffraction patterns show an expansion in the interlamellar d-spacing in the composite samples consistent with intercalation of the clays by the polymers. An interesting feature of these composites is the web or paper like mesh that is formed Details of these characterization of these composite fibers will be presented.







Fibers with diameters of 150-200 nm were found in a mesh of electrospun PEO (1million)/CNTs (1 wt%). The diameters of fibers obtained with 200,000 MW PEO/CNTs (1.5 wt%) composites were ~200 nm. Similar dimensions were obtained for fibers electrospun from the composites containing hectorite A 5 minute deposition produced a mesh with an average thickness of ~3-4 μm thick. Raman spectroscopy, which is extensively used to characterize carbon nanotubes, proved useful in characterizing the composite fibers. Examination of the Raman spectra of the Polymer/CNT composite fibers, it is clear that the carbon nanotubes are present. Raman bands corresponding to nanotubes in the composites are shifted by ~10cm<sup>-1</sup> towards higher wavenumbers. There are also other features in the spectra that are consistant with debundled CNTs.. Similar shifts were also found in the PEO (1 million MW)/10% hectorite/CNT composite fibers. Further details of these results will be discussed. These results show that electrospinning is a useful technique for processing composites of carbon nanotubes with the added advantage of debundling the CNTs during deposition.

# Continuous Processing and Yarn Properties of Electro-Spun Polyacrylonitrile Solution

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# Investigation of Structure-Property Relationship in In-Situ Composite Fibers Based on a Thermotropic Liquid Crystalline Polymer

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This paper is focused on the correlation of morphology with properties of PEN/Vectra blend fibers. Different analytical methods, such as capillary rheology, TGA, DSC, Raman spectroscopy, real-time temperature measurement, SEM and Instron tensile measurement were used to investigate the development of fibril structure in PEN/Vectra blends during melt spinning and the corresponding effect on the properties of PEN/Vectra composite fiber.

Our results showed that PEN/Vectra A950 blends were partly miscible, with miscibility increasing with increased concentration of Vectra in the blends, and that Vectra enhanced the crystallization process of PEN by performing as a nucleating agent. The mechanical properties of PEN/Vectra blend fibers were highly dependent upon the dispersion state and orientation of the Vectra phase.

# **Process Oriented Analysis**

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Process Oriented Analysis is a newly developed set of analysis tools for complex production systems. This methodology was developed by the Institute of Manufacturing Automation and further applied in the education of students and in several industrial projects. Process Oriented Analysis consists of several static and dynamic diagram types. Different to UML, Process Oriented Analysis has a consistent relationship between the diagram types; and additionally the static process-flow-view of the system is provided.

The toolbox of Process Oriented Analysis enables to analyse production systems in a static domain, given by a certain time frame, and in a dynamic domain, where time dependent changes can be monitored. The static diagram types reveal the structure of the system as flows and processes. The diagrams are hierarchically structured on several levels of detail allowing an indepth analysis of complex systems with numerous sub-systems. A system optimisation is possible based on costs or on energy. The cost optimisation is realized by the Value Flow Diagram, which includes calculation of the value of every flow and the value-added of each process. The energy optimisation is achieved by the Resource Flow Diagram, which calculates and balances energy and exergy in order to point out possible savings.

The dynamic diagrams support plant simulation, real time monitoring and control systems, and sustainability models. The dynamic diagrams are consistent with the static diagrams, and simplify program design and coding. A simulation is programmed in order to investigate a production line and scenarios for alternative processing; it is also used to check the performance of machines. Control systems are programmed to enable the set up and operation of production machines. Sustainability models assess the sustainability and efficiency state of a production process, and give recommendations for actions necessary to change to a more beneficial state.

Every diagram type, as well as every program, may be combined with each other in order to configure a suitable instrument for the analysis of a specific system and an individual problem. The acceptance of this graphically based methodology is good among students and professionals, because it enables to understand the function and state of a system quickly and easily. At this point in time a textbook and a software is being developed.

Examples: Flow Diagram of Hemp Fibers

Resource Flow Diagram of a cotton T-Shirt Value Flow Diagram of a weaving mill

State Chart of the M8300 loom

# New Method of CW Protective Composite Development: An Exploratory Study

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The support of the US Army SBCCOM has enabled the initiation of nowovens research program at Texas Tech University. The principal aim of the research effort is to develop nonwoven based chemical warfare protective substrates. A "state-of-the-art" H1 technology needle-punching machinery has been acquired from Fehrer, AG, Austria. Texas Tech University is the *first and only facility* in the US to house the modern needle-punching machinery.

Two different approaches have been followed in the development of the nonwoven based CW protective substrates. The difference in the two methods lies in the nature / structure of the adsorbent layer that is incorporated in between the nonwoven layers. Three layered composite substrates have been developed. The composite substrate consists of:

- (1) prefilter nonwoven layer;
- (2) middle adsorbent layer and
- (3) "next-to-skin" layer

The needle-punching machine has been effectively utilized to develop base substrates from apparel grade polyester fibers. These substrates were produced from polyester fibers (1.5" in length and 1.5 denier) and were processed through a regular needleloom feeder line. The feeder line consists of: a) hopper feeder; b) double cylinder card with 10 carding zones; c) crosslapper. The crosslapper feeds to the H1 technology needle loom.

Polyester needle-punched nonwoven substrates were punched on both sides at a rate of 800 strokes/min. The nanofiber web was then sandwiched between the prefilter and "next-to-skin" layers and was punched at a speed of 800 strokes/min to develop a three-layered composite CW protective substrate.

Figure 1 shows the cross sectional image of nanofiber CW protective composite. As is clearly evident from the image, nanofiber web was successfully incorporated into the web. Due to the limited availability of the material, only tensile strength studies were undertaken so far. Efforts are underway to study the adsorbency characteristics of nanofiber nonwoven CW composite substrate.



Figure 1: Nanofiber based CW Protective Composite: Cross Sectional Image (x150)

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1) Ramkumar, S. S., "Method of Producing Chemical Protective Composite Substrate," US Provisional Patent Application, June 13, 2002.

# Fibers and Films of Polyacryonitrile Acrylic Acid Copolymers M.M. Demir<sup>a</sup>, A. V. Mironov<sup>b</sup>, A. R. Khokhlov<sup>b</sup>, and B. Erman<sup>a</sup>

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Electrospinning is a novel technique for fiber formation and offers an alternative way to the conventional fiber formation process. Main advantage of electrospinning is the possibility of obtaining submicron diameter size fibers. High surface area to volume ratio and high aspect ratio of fibers generate wide application areas from textile to composite reinforcement, wound dressing, catalysis of reaction, etc.

Polyacrylonitrile-acrylic acid (PAN-AA) copolymers form associative gels when dissolved in organic solvent (N,N-dimethylformamide). The reason is the formation of ion pairs between counter ions and negative charges on polymer chains in organic medium, with subsequent aggregation of the ion pairs in ionomeric multiplets.

Nanofibers of PAN-AA copolymers which have various acrylic acid contents (2 wt% -10 wt%) are electrospun from solution. Viscosity of solution is the dominant factor in determining fiber diameter. The diameter of the fibers obtained from PAN-AA copolymer becomes smaller as the AA content increases. The diameter of the fibers obtained from 4.8 wt.% PAN homopolymer is 250 nm on average whereas the same concentration of PAN-AA copolymer of 90 wt% PAN and 10 wt% AA (90:10) generates fibers 20 nm in diameter under 2.2 kV/cm applied electrical field, Fig.1. Thus, it is possible to obtain higher surface area to volume ratio by increasing AA content on polymer chain. Since the viscosity of PAN-AA becomes lower as the AA content increases, the workable concentration interval of polymer solution increases. The upper and lower limits of PAN homopolymer are 2.3 – 10.0 wt %, respectively, whereas the concentration interval of PAN-AA (93:7) is 6.4-14.9 wt.%.

As a result of the electrostatic fiber formation process, a thin and porous film composed of nanofibers is obtained. The same concentration of polymer solution is used to obtain films cast from solution. The area of polymer solution covered on a glass slide and the time needed for the cast films to detach from the glass are recorded. A PAN homopolymer solution covers a smaller area than the same amount of a PAN-AA solution. The presence of AA, as small as 2 percent, increases the area covered three times. Moreover, the detachment time for the cast PAN film from the glass slide in distilled water is 5 hours, whereas for the PAN-AA (98:2) and PAN-AA

(90:10) this time is 1 hour and 10 min, respectively. From the data above, it is easy to say that the surface behavior of copolymer varies with the presence of AA comonomer. Differential scanning calorimeter (DSC), AFM and X-ray meaurements on the cast and electrospun films are carried out to study the influence of the acidic comonomer before and after electrospinning process. The morphology of fibers is examined with AFM (Atomic Force Microscope), OP (Optical Microscope) and SEM (Scanning Electron Microscope).

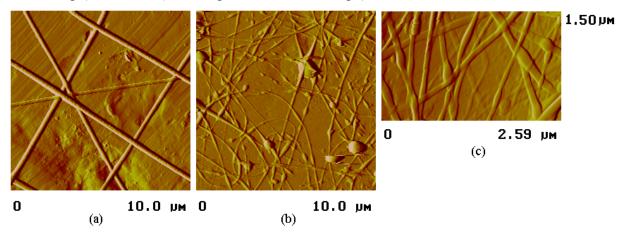


Figure 1. AFM amplitude image of 4.8 wt.% of (a) PAN, (b) PAN-AA (95:5), (c) PAN-AA (90:10) at 2.2 kV/cm applied field.

## **Biographical Information**

Mustafa M. Demir was born in Urfa, Turkey. He developed an interest in science in high school. He received his B.S. in chemistry from Bogazici University in 1999. He then joined the newly founded Sabanci University as a graduate student and received his M.S. in Material Sciences and Engineering in 2001. His research interests range from polymer physics and chemistry to fiber spinning techniques. Currently, he is working for his Ph.D. in Material Science at Sabanci University.

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# Fundamental Investigations on the Electrospinning Process to Make Polymeric Nanofibers

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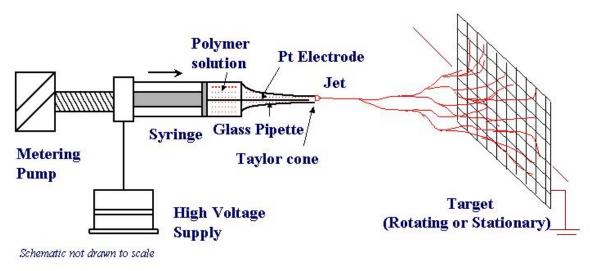


Figure 1. Schematic of the Electrospinning Process

The process of producing thin polymer fibers (50 nm – 1 mm) from polymer solutions by means of electrostatic repulsion is defined as electrospinning. As shown in Figure 1, a typical electrospinning setup consists of a syringe-like apparatus connected to a capillary that contains the polymer solution. A platinum electrode immersed in the solution is connected to a high voltage DC supply. With increasing voltage, a polymer jet is formed and as this jet travels through the air, the solvent evaporates leaving behind a charged fiber that is collected on a grounded target/substrate. Different parameters that affect the morphology of the resulting polymeric nanofibers can be grouped into two categories, namely the process and system parameters. The process parameters that will be discussed in this poster are solution concentration, distance between the capillary-end and the grounded substrate, motion of the substrate, electric potential and flow rate of the polymer solution. Different concentrations (15-25 wt %) of Estane (a segmented ether polyurethane supplied by Goodrich) were prepared in dimethyl acetamide (DMAc). These solutions were then electrospun at different voltages (10-14kV), capillary-screen distances (12-27 cm), flow rates (3-7 ml/h) and different rotational speeds (200-900 rpm) of the substrate.

The effect of one of the system parameters, namely the molecular weight, on the morphology of electrospun fibers will also be discussed. Four grades of PVDF (from 115,000 to 250,000 Mw) were dissolved in DMAc at different concentrations. Electrospun PVDF fibers were obtained at conditions similar to those described above.

Results from preliminary investigations conducted on side-by-side bicomponent fiber electrospinning of PVC and Estane will also be presented and discussed.

#### Biography of the presenting author:

Pankaj Gupta received his Bachelor of Technology (B. Tech) in Textile Technology from Indian Institute of Technology, IIT, Delhi, in 1998 and a M.S. in Polymer Engineering from University of Tennessee in 2000. He joined the Chemical Engineering program at Virginia Tech in Fall 2002 to pursue his doctoral studies. His primary research interests include investigations on structure-property-process relationships in polymers. More recently he has been studying electrospinning to develop materials that could be utilized for novel applications.

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# Contribution to the Understanding of Tribological Mechanisms During an Abrasive or Chemical Wear of Fibrous Structures: Industrial Application to Polyester Wool Fabrics

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The purpose of this work is the permanent, i.e. washing resistant, improvement of the touch of polyester wool elasthan fabrics. Controlled wear have been provided and modifications of the surface states of the worn fabrics have been characterized during a complete tribologic study owing to specific textile apparata. Then, modifications of fabric surface state, transversal compression properties and hairiness have been measured at the different steps of each treatment. The results have permitted to write a matrix formalization of the evolution of tribologic phenomena when the size and density of abrasive particles, the real contact area between particles and the worn fabric, the sliding wear distance and the structure of the worn fibrous structure change.

#### Introduction

Sanding process is well-known in textile industry. The aim of such a treatment is to create a hairiness to confer softness. This type of process is currently used on cotton fabrics. However, adapting conditions of wear for a specific treated fabric is the major difficulty of this type of treatment. The control of sanding is today totally empiric in industrial processes because there is a total absence of clear-cut definitions. To produce sanded fabrics, it is so necessary to make many trials on a sanding machine with the same fabric. The industrial optimization of the sanding process is then becoming very complex. It is very important to consider the complex fibrous structure, which will be worn. The influence of abrasive characteristics on the wear mechanisms is today unknown even if, with extreme wariness, some researchers stipulate that micro abrasive particles provide fine hairiness on the surface of the fabric whereas macro grits induce longer piles. The aim of this work is to define, quantify and compare the tribologic phenomena, which appear on a fibrous structure during wear process when the topography of abrasive papers changes.

#### **Apparatuses**

To understand the improvement of touch, tribologic characterization of fabrics is necessary. Since Amontons law [1] was enunciated and extended to fibrous structures ( $F=\alpha Nn-1$ ), notion of real contact area (compared to the apparent contact area) during friction on textile surfaces have been taken a great importance within the characterization of the surface state. Many apparatuses had been developed in

order to measure static and dynamic friction coefficients and stick-slip traces on textiles [2; 3; 4]. Parameters like material structure, type of yarn and weave of fibrous structures induce fundamental variations of friction behavior. Nevertheless the friction coefficient can not be considered as the single parameter that characterizes the surface state of a fabric. In that way, Bueno *et al.* had developed specific apparatuses [5; 6;7]. The first one is a multi-directional tribometer (figure 1) that measures the friction and the roughness of a fabric. This latter is stretched on a rotative plate. A cylindrical probe (diameter of 0,5 mm) is rubbed on the fabric surface. An accelerometer is fixed on this probe and measure vibrations due to its penetration in the fabric structure.

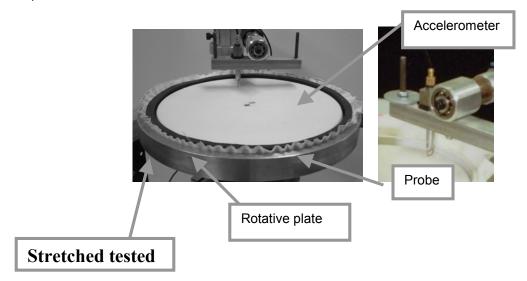


Figure 1: Multi-directional Tribometer

Owing to a FFT analysis of the temporal signal, a spectrum is computed on it, the peaks energy is dependent on the magnitude of the structure relief. For example, in the specific case of a satin structure, the long warp floats of 4 make the weft yarns totally sunken and then, the rib effect only can induce the presence of a frequency peak. The autospectrum (power spectral density) has then a unique peak, from which the spectral energy density is extracted (figure 2).

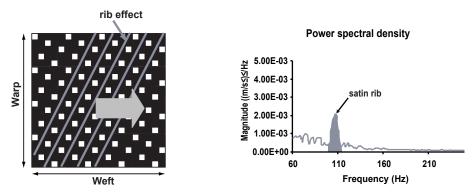


Figure 2: Spectral analysis of the satin structure

The second apparatus that was used is the KES – FB3 (Kawabata Evaluation System of Fabrics, compression modulus). It measures the compression behavior of fabrics under low stresses during a compression – decompression cycle. A cylindrical pin, equipped with a strength sensor, compresses the fabric until 50 cN/cm² and measures the strength versus thickness of this fabric. A typical curve is obtained and two values are used during this work: Thickness under 50 cN/cm² ( $T_m$ ) and energy loss during the cycle ( $E_m$ ), given by the following equation (1):

$$Em = \int_{0}^{50cN} F_{1}(t)dt - \int_{50cN}^{0} F_{2}(t)dt$$
 (1)

where  $F_1$  measures strength during the compression part of the cycle and  $F_2$ , the strength during the decompression part of the cycle.

The third apparatus is the optical pilemeter developed by Bueno *et al* [20]. The principle is given on the figure 3. A laser beam illuminates the tested fabric and a photodiode measures the energy due to the fabric hairiness. The measured value, which is called Q, is dependent on the total length of pile (or hairiness Quantity) on the fabric surface.

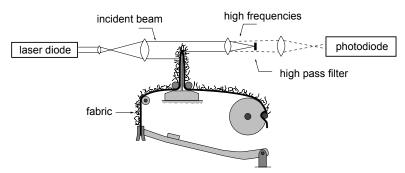


Figure 3: Optical pilemeter

The tribologic comprehension of modifications occurring on textile surface after chemical or mechanical wear can now start. It is important to make clear that the present work takes place in an industrial context where wearing process of fibrous structures is totally empirically controlled.

## **Experimental**

Two different polyester wool fabrics (Plain weave and satin) made of the same yarn in warp and weft have been worn and studied. The characteristics of these fabrics are showed in table 1.

Weave	Warp yarn density/cm	Warp yarn density/cm	Composition	
Plain weave	25	25	50 % polyester / 50 % wool	
Satin of 5	27	24	50 % polyester / 50 % wool	

Table 1: Fabric characteristics

## Abrasive wear of fibrous structures

In industry, the main problems occurring during abrasive wear of fabrics are the consequence of a lack of definition of the optimal conditions in which sanding must occur. The aim of this study have been to control and to modify parameters of wear in order to understand mechanical phenomena that appear on the surface of the worn fabric. Such a study led rapidly to an enormous consumption of fabric an then, a high cost. To solve this problem, a new lab sanding machine was developed in order to approach the industrial sanding conditions (figure 4). On this machine, the fabric is carried and stretched between two pairs of cylinders (1) in front and in back of the machine. The fabric is then rubbed around the rotative abrasive cylinder (2) according to an adjustable  $\alpha$  sanding angle. This prototype allows to wear small bands of fabric (a few meters long and 30 cm wide) under very versatile conditions. It is for example easy to modify the type of abrasives, the sliding length, the production speed or the speed of the sanding head.

For this study, we decided to fix several parameters like speed of the rotative abrasive head, tension and speed of the fabric. The parameters that we decided to modify were the type of emery papers which are called by their F.E.P.A (European Federation of abrasives papers producers) normalized name as shown in table 2, the sliding distance (form 1 to 4 passages on the machine) and the structure of the worn fabrics (Polyester wool elasthan plain weave and satin described in table 1).

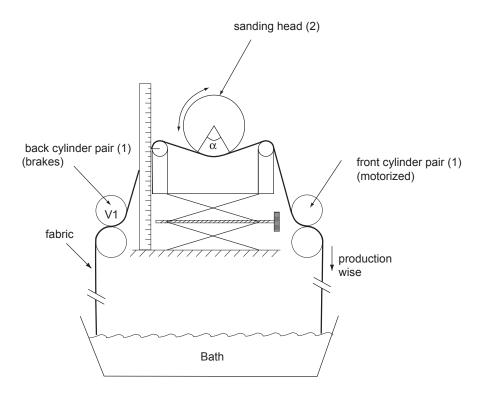


Figure 4: lab sanding machine

# **Theory**

The limited definitions given by the F.E.P.A. norm can not be sufficient to permit a real comparison of their action during the wear process. It was decided to make profilometric measures on each of the used paper in order to define roughness (Ra), skewness ( $\sigma$ ) and density of particles on the surface of the papers. These characteristics are shown in table 2.

	Particles size (μm)	Ra (µm)	σ (μm)	Particle density /mm²
P 800s	21,8±1	5,5	7,3	706
P 1000s	18,3±1	5,3	7,0	1280
P 2000s	10,3±1	4,4	5,6	1440

Table 2: F.E.P.A. Characteristics

# Tribologic phenomena

Owing to MEB photographies, tribologic wear phenomena have been defined at the yarn and fiber scales. In the case of satin, weft in totally sunk in the structure. Then phenomena can only occur in warp wise. At the opposite, in the case of plain weave fabric, both warp and weft are worn during the sanding process, the phenomena can occur in the two directions. These tribologic phenomena are:

## At the fiber scale

 $W_f$ : Wear of the surface of fibers, held by its two extremities on the surface of the fabric. This phenomenon induces the creation of a micro-groove on the fiber (figure 5).

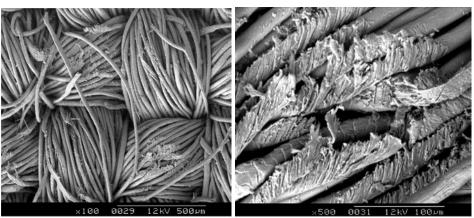


Figure 5: Wear of the surface of fibers

 $W_{ef}$ : Wear of emergent fibers ( $W_{ef}$ ), held only by one of its two extremities on the surface of the fabric (hairiness) (figure 6).

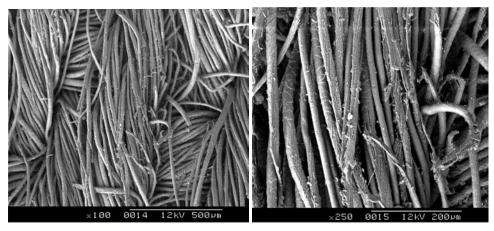


Figure 6: Wear of emergent fibers

# At the yarn scale

Fiber break (B) due to the achievement of the wear  $W_f$  (figure 7). This phenomenon induces the creation of a meso-groove on the yarn. Partial extraction ( $E_p$ ) of fibers from the yarn on the surface of the fabric (figure 8). This phenomenon results from a displacement of material.

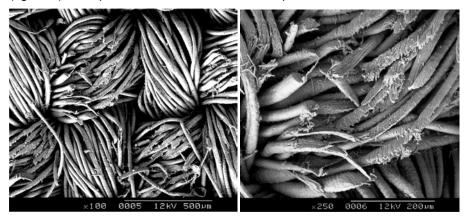


Figure 7: Breaks of fibers

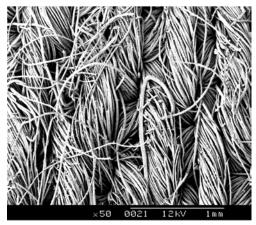


Figure 8: Partial extractions of fibers

#### Matrix formalization

The appearance of each of those phenomena induces different variations on the surface state of the worn fabric. Thus, a matrix, showing the evolution of each phenomenon along the successive sanding passages has been written. In our case, this matrix has the following form shown at the relation (2):

$$\begin{pmatrix} \mathbf{W}_{f} \\ \mathbf{W}_{ef} \\ \mathbf{B} \\ \mathbf{E}_{p} \end{pmatrix} = \begin{pmatrix} \mathbf{\alpha}_{W_{f}1} & \mathbf{\alpha}_{W_{f}2} & \mathbf{\alpha}_{W_{f}3} & \mathbf{\alpha}_{W_{f}4} \\ \mathbf{\alpha}_{W_{ef}1} & \mathbf{\alpha}_{W_{ef}2} & \mathbf{\alpha}_{W_{ef}3} & \mathbf{\alpha}_{W_{ef}4} \\ \mathbf{\alpha}_{B1} & \mathbf{\alpha}_{B2} & \mathbf{\alpha}_{B3} & \mathbf{\alpha}_{B4} \\ \mathbf{\alpha}_{E_{p}1} & \mathbf{\alpha}_{E_{p}2} & \mathbf{\alpha}_{E_{p}3} & \mathbf{\alpha}_{E_{p}4} \end{pmatrix}$$

$$(2)$$

where the coefficients  $\alpha_{ph,i}$  quantify the evolution of the phenomenon "ph" (W<sub>f</sub>, W<sub>ef</sub>, B or E<sub>p</sub>) between the passages i and i+1 on the lab sanding machine. Owing to this formalization, the individual evolution of each phenomenon can be represented on the same graph for each grit, passage after passage.

#### Results and discussion

#### Influence of the worn structure

The main results (figure 9) display that size and density of particles on the surface of the abrasive papers have to be considered simultaneously to understand the occurring of each phenomenon. For a same type of fiber, the results show that the macro-grits (P800 and P1000) wear at the yarn scale opposite to microgrits (P2000) that wear at the fiber scale. Although, the difference between wear mechanisms are sometimes difficult to understand since all the phenomena occur probably simultaneously, i.e. on a float, fibers can be broken whereas on another float, fibers are only worn. This problem induce a global non homogeneity of the surface states difficult to analyze with our method. The particular case of the P800d abrasive process is difficult to analyze, more details are given in the manuscript. However, the particular surface topography of this kind of abrasive paper seems to have a significant influence on the wear process. Moreover, the "island in the sea" structure seemed to induce wear at different scales. In facts, whereas Island can be considered as big particles able to break fibers, the fins diamond particles have the same wear behavior than those of P800s.

It is possible to show Important points as following:

- Considering the ratio size of particles / diameter of the worn,
- Considering the density of particles on the abrasive paper.

Density of particles have a significant importance on the real contact area between the abrasive paper and the worn structure. These real contact area had been approximated owing to the hertz theory. Then, partial extraction of fibers after the first sanding passage can be observed for the P2000 abrasion process even if the particle are too small to scratch the worn fabric. That means that adhesion is sufficient to extract some fibers held by the structure, at the surface of the fabric.

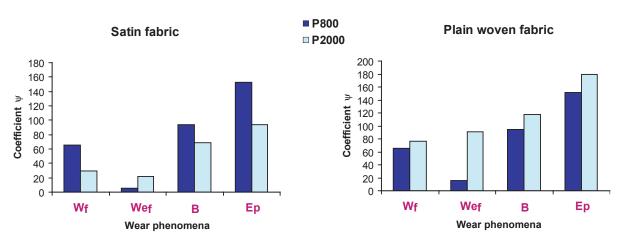


Figure 9: Evolution on tribologic phenomena after 4 passages on the lab sanding machine.

#### Conclusion

This work has made possible to propose sanding treatments on polyester / wool fabrics that were previously considered as impossible to control in industrial conditions. For such a treatment, tribologic phenomena have been defined in order to understand the wear mechanisms occurring on the surface of the worn fabrics. Moreover, a matrix formalization had been developed and propose a quantification of the evolution of each wear phenomenon:

At the fiber scale: Wear of the surface of the fibers (W<sub>f</sub>) and on emergent piles (W<sub>ef</sub>)

At the yarn scale: Break (B) and partial extraction of the fibers (E<sub>p</sub>)

Then, when the size of particles is equivalent to the fiber diameter, yarn scale phenomena are the most significant. On the opposite, when the size of particles decrease, fiber wear scale phenomena are more and more apparent. However, density of particles increases and adhesion occurs because of the real contact area that increase between the particles and the worn fabrics. Moreover, the length of the worn float seems to be an essential parameter to provide a real improvement of softness. Thus, abrasion of the plain weave fabric (short float) induces the creation of a rigid and short hairiness which is unpleasant to touch. On the opposite, abrasion of satin floats creates long worn piles and gives the sensation of raised fabrics whereas abrasion with small particles induces the appearance of a very fine and short hairiness that remind micro-fibers sanded fabrics. This work shows that structure, fiber diameter and abrasive papers used are important parameters to sand fabrics in the best conditions.

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